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Laboratory Evaluation of Dust and Dust Lead Recoveries for Samplers and Vacuum Cleaners

Volume II: Appendices from the Quality Assurance Project Plan

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LABORATORY EVALUATION OF DUST AND DUST LEAD RECOVERIES FOR SAMPLERS AND VACUUM CLEANERS

VOLUME II: APPENDICES FROM THE QUALITY ASSURANCE PROJECT PLAN

Technical Program Branch
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Office of Pollution Prevention and Toxics
Office of Prevention, Pesticides, and Toxic Substances
U.S. Environmental Protection Agency
Washington DC 20460

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1 INTRODUCTION

This project to expand the knowledge on household dust testing methods was undertaken by the U.S. Environmental Protection Agency (EPA) as part of a major national effort to address the public health issue of childhood lead poisoning. The effort was given impetus by the CDC's statement on lead poisoning, which reduced the level of concern for blood lead levels from 25 micrograms/deciliter (µg/dl) to 10 µg/dl. It has also been given impetus by the passage of the "Residential Lead-Based Paint Hazard Reduction Act of 1992," also known as "Title X." In response to Title X, EPA is proceeding towards the development of health-based standards for house dust lead levels. To do this, appropriate methods for sampling house dust are needed. As part of this effort, numerous questions about house dust sampling have risen. This study was designed to address some of these questions.

This project was undertaken by the EPA Office of Pollution Prevention and Toxics (OPPT) to evaluate house dust sampling methods and to assess the efficacy of typical household vacuuming on removing leaded dust from residential surfaces. Dust-lead sampling results from the National Survey of Lead-Based Paint in Housing (HUD National Survey) are reexamined, based on new information collected in this study about the performance of the dust sampler used during that survey.

Two standardized laboratory testing procedures were developed for this study. The first procedure was designed to characterize the performance of house dust samplers. The second was designed to evaluate how well commercially available vacuum cleaners collect dust from various surface types. Three vacuum sampling methods and one wipe sampling method were tested by the first procedure. These methods included the "Farfel modified" High Volume Small Surface Sampler used in the Baltimore Repair and Maintenance study (called the BRM sampler is this report), the Comprehensive Abatement Performance Study (CAPS) cyclone sampler, the Blue Nozzle sampler, and the Department of Housing and Urban Development's (HUD) wipe sampling method. All of these sampling methods have been used in previous EPA/OPPT studies. The second procedure was used to characterize four commercially available household vacuum cleaners ranging in price from $120 to $800. The most expensive vacuum cleaner was equipped with a high efficiency particulate air (HEPA) filter. The protocols for both testing procedures included using real house dust sieved into six particle size classes ranging from 0 to 2,000 microns in size. The dust was applied to five substrates commonly encountered inside a residence: tile, wood flooring, linoleum (sheet vinyl), upholstery, and carpet.

A secondary purpose of the project was to assess the amount of dust exhausted into the air while dust is being vacuumed. The Federal government has concerns that routine vacuuming of highly lead-contaminated dust may create unseen health hazards by polluting the air with lead particles. Lead abatement specialists use vacuum cleaners equipped with a HEPA filter to clean up lead-contaminated dust. The HEPA filters prevent fine lead particles from escaping the vacuum cleaner through the exhaust and, thus, prevent a potential airborne lead hazard. While vacuum cleaners fitted with

HEPA filters are available, they usually are expensive and not readily accessible to the general public, although the situation is improving. This project measured the recovery and exhaust emissions of lead dust in a laboratory setting by four different vacuum cleaners currently available for household use. One of these vacuum cleaners was equipped with a HEPA filter.

The sampling and analysis procedures and the project organization are described in the Quality Assurance Project Plan for the Wipe and Vacuum Study (QAPjP), Revision 1 dated September 24, 1993. The protocols for collecting and handling household dust used in the tests, preparing the substrate sections for the tests, operating the samplers and vacuum cleaners, and performing various steps for the analysis of lead are contained the QAPjP appendices.

The study report is divided into two volumes. Volume I presents the background, methods, and study results. For readers interested in the specific sampling and analysis procedures or those interested in replicating the procedures, Volume II contains the appendices from the QAPjP which describe the sampling and analysis procedures. The Table of Contents lists the various appendices. In the original QAPjP, the BRM sampler was referred to as the HVS3 sampler. The terminology was changed for this report to better describe the sampler. In these appendices the term HVS3 has been changed to BRM except on copies of the data collection sheets used during the study. Any such reference to HVS3 refers to the BRM sampler, the sampler used in the Baltimore Repair and Maintenance study.

APPENDIX A

PROTOCOL FOR COLLECTING AND HANDLING USED HOUSEHOLD VACUUM CLEANER BAGS

PROTOCOL FOR COLLECTING AND HANDLING USED HOUSEHOLD VACUUM CLEANER BAGS

1.0 INTRODUCTION

This protocol provides instructions on how to obtain and prepare household dust to perform the tests according to the QAPjP. Based on the results from the National Survey, the assumption is made that lead levels in household dust correlate with the age of the dwelling. Thus, household dust vacuum bags will be collected from homes built in or after 1983 (newer homes) and in or before 1962 (older homes). All three organizations involved in this task participate in the collection.

2.0 COLLECTION PROCEDURE

- Within each organization (EPA, MRI, and Westat), distribute kitchen trash bags with the following instructions to volunteer staff in their organizations:

  - Put (preferably full) used household vacuum bag (bag and dust) in trash bag. Do this carefully so that the dust from the vac bag does not blow out into the trash bag.
  - Close trash bag with twist tie.
  - Indicate on enclosed label whether home was old (1962 or earlier) or new (1983 or later). Attach label to trash bag.
  - Bring bag to designated area.

- Collect the vacuum bags with their dust content at each organization. Each vacuum bag will be in a separate kitchen bag and tagged as to the age of the home (older or newer).

3.0 STERILIZATION PROCEDURE

- After collection, package the bags in cartons at each organization and ship via Federal Express to the following address:

Neutron Products
Attn: Ms. Geraldine Barrett
22301 Mount Ethraim Road
P.O. Box 68
Dickerson, MD 20842
Telephone: (301) 349-5001

- At Neutron Products, have the bags sterilized by radiation, according to the procedure developed by RTI and followed by ORD and NIST.

- After sterilization, have Neutron Products ship the cartons from all three organizations back to MRI by Federal Express.

APPENDIX B

PROTOCOL FOR SIEVING HOUSEHOLD DUST

PROTOCOL FOR SIEVING HOUSEHOLD DUST

1.0 INTRODUCTION

The household dust is contained in the original vacuum bags collected by EPA, MRI, and Westat volunteers. Each bag has been sterilized by radiation and shipped back to MRI (see Appendix A). The bags will each be tagged as to the two categories of homes: built in or before 1962 (older homes), or built in or later than 1983 (newer homes). This protocol provides instructions to sieve the dust in both categories into six particle size classes. The sieved fractions from each category will be composited and used as the test dust for the laboratory testing under this project.

2.0 EQUIPMENT AND SUPPLIES

- Vacuum dust bags

- Six 8-in stainless steel sieves, soldered with 100% tin, lead-free, as follows:

    - Less than 2,000 μm: Sieve No. 10
    - Less than 250 μm: Sieve No. 60
    - Less than 212 μm: Sieve No. 70
    - Less than 150 μm: Sieve No. 100
    - Less than 106 μm: Sieve No. 140
    - Less than 53 μm: Sieve No. 270

- 8-in pan with lid

- Mechanical shaker sieving device

- Plastic containers for sieved dust (minimum of 12)

- Balance to weigh full bags and to weigh sieved dust in containers

- Balance to weigh samples of sieved dust

- Soft brush

- Plastic trash bags

- Small plastic bottles for samples of sieved dust (24)

- Ziplock plastic bags

3.0 PREPARATION PROCEDURES

- Weigh the dust bags separately in each of the two categories and record total weight for each category on data entry forms at the end of this Appendix B.

- Label containers as follows, where "high" corresponds to dust samples from older homes and "low" corresponds to dust samples from newer homes:

1. High: 250-2,000 μm
2. High: 212-250 μm
3. High: 150-212 μm
4. High: 106-150 μm
5. High: 53-106 μm
6. High: less than 53 μm
7. Low: 250-2,000 μm
8. Low: 212-250 μm
9. Low: 150-212 μm
10. Low: 106-150 μm
11. Low: 53-106 μm
12. Low: less than 53 μm

- Weigh at least 12 empty plastic containers (tare weight) in which sieved dust composites will be stored, and record weights on data entry forms at the end of this Appendix B.

4.0 SIEVING PROCEDURE

NOTE: Always keep the two categories of dust bags (older homes and newer homes) separated. Start with the bags collected in the newer homes to minimize lead contamination.

- Clean the sieves with compressed air and/or a soft brush. Material lodged in the sieve openings or adhering to the sides of the sieves should be removed (if possible) without handling the screen roughly.

- Nest the sieves in decreasing order in the sieving device, with pan at the bottom. (Sieving device to be located in a hood.)

- Empty dust bag(s) from one category homes onto top sieve. Empty as many bags as necessary to have sufficient dust sample on top sieve. Discard empty vacuum bags in trash bag.

- Sieve for 5 minutes. Redistribute material on top of sieve and sieve for 5 more minutes.

- Discard material from top sieve in trash bag.

- Collect the dust samples from all but the top sieve and from the pan and transfer to appropriate labelled containers.

- Repeat sieving procedure with additional bags from one home category until enough dust is obtained in each size category are exhausted. Use additional containers if necessary.

- Weigh containers when full or when sieving is completed and record total weight of each container on weighing form (see Section 3.1 of QAPjP).

- Clean the sieves with compressed air and/or a soft brush, whenever necessary during sieving operations.

- Repeat dust sieving, dust collection, weighing and recording for the second category of dust bags (older homes).

- For each category of dust bags, sieve additional bags until enough dust has been obtained for the tests. For dust from older homes, start with those bags which can be identified as coming from the oldest homes, if possible.

5.0 PREPARE SAMPLES FOR ANALYSIS

- Obtain 24 small, clean plastic bottles for samples to be analyzed. Place a bar code label on each bottle and an identical label on the weighing form. Also, place a handwritten label on each bottle that identifies the test category and class (new homes/old homes, size class).

- Weigh the empty bottles (with lid and labels). Record weight on data form.

- Thoroughly mix the contents of each container of dust.

- Take 10 small subsamples (approx. 70 mg each) from each container of dust (or from all containers in which that dust category is stored). Composite these subsamples into the sample bottle for that category. Repeat this subsampling once to obtain duplicate samples.

- Reweigh the sample bottles containing the composite samples (total of 24 samples, including duplicates). Record weight on data form, and test number per attached test sequence.

- Place each sample bottle in a ziplock plastic bag. Submit samples for Pb analysis.

The Data Entry Sheet for Initial Analysis of Sieved Dust is shown following paragraph 6.0

6.0 COLLECTION OF SIEVED DUST SAMPLES BEFORE TESTS, AND WEEKLY

Samples of sieved dust will be taken for Pb analysis just prior to the tests and weekly thereafter (total of 12 samples each week). These will be taken in a manner that duplicates the application of dust onto substrates. Approximately 0.678g of each category of dust (size and Pb concentration) will be taken, and using the dust application device, the dust will be collected on a plastic sheet. Dust on the plastic sheet will then be transferred into a weighted sample bottle and analyzed for Pb. Data forms for these samples, and the test number sequences are found following this paragraph.

Data Form for
Sieving of Dust Bags

Date ______________________
Operator ______________________

Dust Bags from ______________ (old or newer homes)
Number of Dust Bags Sieved __________
Total Weight of Bags Sieved __________
(in grams)

Weight of Dust Recovered (by size):

	≤ 53 μm	53-106 μm	106-150 μm	150-212 μm	212-250 μm	250-2,000 μm
Final Wt (g)						
Tare Wt (g)	____	____	____	____	____	____
Net						

Reviewed by ______________________

Data Entry Sheet
for
Initial Analysis of Sieved Dust

Procedure

Test No. ____________

Thoroughly mix each container of dust from new homes
Tare weigh 12 sample bottles, with bar code labels
Take 10 random subsamples of about 70 mg each from one container, and combine in first sample bottle
Reweigh sample bottle after taking 10 subsamples
Repeat preceding step (i.e., duplicate sample)
Repeat the above steps for each size of dust (total of 12 samples including duplicates)
Using new data sheet, repeat all the above for dust from the older age of homes

Date ____________
Operator ____________
Dust Type ____________ (old or new homes)

Sample Weight (Balance No. ____________)

Sample Weight (Balance No. ____________)

≤ 53 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

≤ 53 μm (dup)

Final Wt
Tare Wt ________
Net

Bar Code Label

53-106 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

53-106 μm (dup)

Final Wt
Tare Wt ________
Net

Bar Code Label

106-150 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

106-150 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

150-212 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

150-212 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

212-250 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

212-250 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

250-2000 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

250-2000 μm

Final Wt
Tare Wt ________
Net

Bar Code Label

	Date
Samples Relinquished by: ____________	______
Samples Received by: ____________	______
Reviewed by: ____________	______

Data Entry Sheet
for
Pretest and Weekly Analysis of Sieved Dust

Test No. ________________
Date ________________
Operator ________________
Dust Type ________________
(old or new homes)

Procedure

Collect sample of each dust size, from new homes, as follows:

- Take approximately 0.678 g from dust container
- Deposit dust through sieve, onto plastic sheet, to simulate depositing of dust on substrate
- Determine weight of dust deposited
- Transfer dust on plastic sheet into labeled and weighed sample bottle
- Determine weight of dust sample in sample bottle
- Repeat all the above for each particle size
- Using new data sheet, repeat all the above for dust from older homes

	Dust Applied (gm) (Balance No. _______)			Dust Sample (gm) (Balance No. _______)		
Size	Total Wt	Final Wt	Net Wt	Total Wt	Tare Wt	Net Wt
> 53 µm	______	______	______	______	______	______	Bar Code Label
53-106 µm	______	______	______	______	______	______	Bar Code Label
106-150 µm	______	______	______	______	______	______	Bar Code Label
150-212 µm	______	______	______	______	______	______	Bar Code Label
212-250 µm	______	______	______	______	______	______	Bar Code Label
250-2000 µm	______	______	______	______	______	______	Bar Code Label

Samples Relinquished by: ____________________
Samples Received by: ____________________
Date: ____________________

Reviewed by ____________
Date ____________

Test Sequence for Samples of Dust

Lead concentration in the source dust
Table shows each sample of dust collected for analysis

Test number	Substrate sample: Substrate	Dust Loading	Lead Conc	Particle size	New bag	Dust deposited (mg)	Vacuum	Team	time vacuumed	dust collected (mg)	dust sent for analysis (mg)	lead amount (ug)	Date Collected	Time collected	Target Dust deposited (g)
601	Sample of dust for analysis		Low	<53									Prior to testing		0.678
602	"		Low	53-106											0.678
603	"		Low	106-150											0.678
604	"		Low	150-212											0.678
605	"		Low	212-250											0.678
606	"		Low	250-2000											0.678
607	Sample of dust for analysis		High	<53											0.678
608	"		High	53-106											0.678
609	"		High	106-150											0.678
610	"		High	150-212											0.678
611	"		High	212-250											0.678
612	"		High	250-2000											0.678
621	Sample of dust for analysis		Low	<53								Prior to testing (duplicate)			0.678
622	"		Low	53-106											0.678
623	"		Low	106-150											0.678
624	"		Low	150-212											0.678
625	"		Low	212-250											0.678
626	"		Low	250-2000											0.678
627	Sample of dust for analysis		High	<53											0.678
628	"		High	53-106											0.678
629	"		High	106-150											0.678
630	"		High	150-212											0.678
631	"		High	212-250											0.678
632	"		High	250-2000											0.678
641	Sample of dust for analysis		Low	<53								After dust preconditioning, before tests			0.678
642	"		Low	53-106											0.678
643	"		Low	106-150											0.678
644	"		Low	150-212											0.678
645	"		Low	212-250											0.678
646	"		Low	250-2000											0.678
647	Sample of dust for analysis		High	<53											0.678
648	"		High	53-106											0.678
649	"		High	106-150											0.678
650	"		High	150-212											0.678
651	"		High	212-250											0.678
652	"		High	250-2000											0.678

Lead concentration in the source dust (continued)
Table shows each sample of dust collected for analysis

	Substrate sample														
Test number	Substrate	Dust Loading	Lead Conc	Particle size	New bag	Dust deposited (mg)	Vacuum	Team	time vacuumed	dust collected (mg)	dust sent for analysis (mg)	lead amount (ug)	Date Collected	Time collected	Target Dust deposited (g)
661	Sample of dust for analysis		Low	<53							With first weeks batch of samples				0.678
662	"		Low	53-106											0.678
663	"		Low	106-150											0.678
664	"		Low	150-212											0.678
665	"		Low	212-250											0.678
666	"		Low	250-2000											0.678
667	Sample of dust for analysis		High	<53											0.678
668	"		High	53-106											0.678
669	"		High	106-150											0.678
670	"		High	150-212											0.678
671	"		High	212-250											0.678
672	"		High	250-2000											0.678
681	Sample of dust for analysis		Low	<53							With second weeks batch of samples				0.678
682	"		Low	53-106											0.678
683	"		Low	106-150											0.678
684	"		Low	150-212											0.678
685	"		Low	212-250											0.678
686	"		Low	250-2000											0.678
687	Sample of dust for analysis		High	<53											0.678
688	"		High	53-106											0.678
689	"		High	106-150											0.678
690	"		High	150-212											0.678
691	"		High	212-250											0.678
692	"		High	250-2000											0.678
701											Continue until the tests are complete ...				
...															

IF there are 10 batches of samples where will be 156 dust analyses on these dust samples, with 9 grams of dust required for each size and lead level class

APPENDIX C

PROTOCOL FOR GRINDING DUST INTO CARPET

PROTOCOL FOR GRINDING DUST INTO CARPET

1.0 INTRODUCTION

Some tests on this study require grind-in of test dust into the carpet substrate. Further, the area of the substrate on which the test dust is to be applied and ground-in will depend on whether the test is to be done using a research vac (1 ft^2) or a Housevac (6.78 ft^2). In either case, the grind-in will be done using the same grind-in tool as listed below, which was built in accordance with that in ASTM F 608-89.

2.0 EQUIPMENT AND SUPPLIES

- Grind-in tool
- Small paintbrush
- Templates (1 and 6.78 ft^2)
- Wide masking tape
- Stopwatch
- Carpet

3.0 PROCEDURE

This procedure is to be used in part of the dust preconditioning of carpet/upholstery substrates and as part of housevac and sampler tests. Refer to those appendices (App D, I, P, Q, and R) for test sequences and data forms.

3.1 Preparation for Grind-in

- Weigh out the amount of test dust to be used, depending on the test to be conducted:

Test of research vac:	100 mg or 400 mg	(±10 mg)
Test of Housevac:	678 mg or 2,712 mg	(±10 mg)

- Be sure the test dust used corresponds with that specified for the tests [correct particle size range and correct Pb content (high or low Pb)]. Transfer the weighed

amount of dust into a shaker or other suitable device for distributing dust onto the carpet. Weigh the device and dust together, and record as total weight.

- Place the template on the carpet. Use masking tape to mark the test area. Distribute the dust as evenly as possible over the test area, inside the template. Remove the template, being careful to tap and brush the template so that any dust adhering to the template falls back onto the test area.

- Distribute dust onto substrate.

- Reweigh device with any remaining dust. Calculate net weight of dust deposited.

3.2 Grind-in

- Embed the test dust into the carpet using the grind-in tool. Perform the grind-in by using a dragging motion in both directions, alternating directions forward and back. <u>Do not</u> drag the tool in one direction and push it back the other direction (even though this would be more convenient). Hold the handle near a 45-degree angle when dragging the tool across the test section.

- Drag the tool over the test area, exactly 30 strokes, using a uniform movement and stroke time of 2.5 sec per stroke (i.e., 30 strokes over a total time of 75 sec.) (A movement in one direction is one "stroke.") Be sure the tool covers the entire test area on every stroke (i.e., ends of the tool extend over both sides of the test area at all times).

- After completing the grind-in, use the small paintbrush to brush any material adhering to the tool back onto the test area. Remove the grind-in tool from the test locale, and clean it off with compressed air, if necessary, prior to the next use.

- Clean the template with compressed air.

4.0 DEVIATIONS FROM PROTOCOL

Every attempt shall be made to follow this protocol. Deviations from the protocol may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual (or unanticipated conditions).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the team leader and the MRI Work Assignment Leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation shall be recollected in accordance with the specified protocol.

Deliberate deviations from the sampling protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be expedited. The MRI Work Assignment Leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals.

APPENDIX D

PROTOCOL FOR CONDITIONING CARPET AND OTHER SUBSTRATES

PROTOCOL FOR CONDITIONING CARPET AND OTHER SUBSTRATES

1.0 INTRODUCTION

Tests on this study involve several different substrates (e.g., carpet, upholstery, wood). Some preconditioning of each of these substrates is necessary prior to their use in the tests, especially carpets. For example, carpet must be vacuumed several times before use in any tests, because a substantial amount of carpet fibers are collected when a new carpet is first vacuumed, which would interfere with determination of recovery of dust applied to the carpet.

After preconditioning, one section of each substrate will be used in all tests, so long as those tests involve the same dust loading (i.e., 100 or 400 mg/ft^2) and the same concentration of Pb in the dust (i.e., low Pb or high Pb). Therefore, four separate sections of each substrate will be required, identified for use in the following tests:

- Low loading/low lead
- Low loading/high lead
- High loading/low lead
- High loading/high lead

Preconditioning procedures for each substrate are described below.

2.0 EQUIPMENT AND SUPPLIES

- Four sections of each substrate (72 x 27 in.)
- Grind-in tool
- Template (18 x 54 in.)
- Duct tape
- Dust to be applied
- Housevacs with attachments for each substrate
- Stopwatch

3.0 PROCEDURES

Procedures are described below for each of the substrates to be tested:

- Carpet
- Carpet with grind-in
- Upholstery
- Wood
- Tile
- Linoleum

3.1 FIBER PRECONDITIONING OF CARPET AND UPHOLSTERY SUBSTRATES

Fiber preconditioning of carpet and upholstery substrates will be carried out in accordance with the test sequence and data form attached at the end of this appendix. For each section of substrate, this involves vacuuming for 5 min. with each of the four Housevacs in sequence (starting with a specific Housevac for each section of substrate as specified in the test sequence). Vacuuming with each Housevac in sequence, continue as many times as is necessary to achieve the criteria given below. The procedure for this fiber preconditioning of each section of substrate is as follows:

a. Record data using data entry forms attached at the end of this appendix.

b. Use a new bag for each Housevac each morning.

c. Tare weigh bag prior to first use:
   Run free for 5 min, cool 2 min, brush* and record weight after 1 more min
   *(refers to brush of bag with anti-static brush)

d. Vacuum substrate section for 5 min, starting with the Housevac identified in test sequence.

e. Reweigh bag after vacuuming for 5 min: (Cool 2 min, brush and record weight after 1 more min)

f. Proceed with 5 min vacuuming with each Housevac per test sequence.

g. Continue 5 min vacuumings until substrate has been vacuumed a total of 20 times, or until weight gain reaches 20 mg or less for 4 consecutive vacuumings.

3.2 DUST PRECONDITIONING OF ALL SUBSTRATES

Dust preconditioning of all substrates will be carried out in accordance with the test sequence and data form attached at the end of this appendix. For each section of substrate, this involves several applications of dust of different sizes, and vacuuming for 40 sec using a different Housevac each time. The procedure for the dust preconditioning of each substrate is as follows:

a. Mark test area on carpet using template and duct tape.
b. Use a new bag in each Housevac at beginning of each day.
c. Perform the tests according to the test sequence. Be sure the substrate is properly identified for the specific dust loading (100 or 400 mg/ft^2) and specific Pb concentration (low or high), for which it will be used in all subsequent testing of Housevacs and samplers.
d. Deposit required amount of dust having the particle size specified in test sequence. Determine actual weight of dust deposited.
e. Grind in dust, if applicable (per Appendix C).
f. Determine tare weight of each bag before each use:
    Run free for 40 sec, cool 2 min, brush and record weight after 1 more min
g. Vacuum for 40 sec with the Housevac specified in the test sequence.
h. Record time of the vacuuming.
i. Reweigh bag (cool 2 min, brush, and record weight ater 1 more min).
j. Repeat tare-vac-reweigh (steps d-i above) using the next Housevac and particle size designated in the test sequence. Continue until all tests shown in the test sequence are completed for that substrate section.
k. Vacuum wand and brush on all Housevacs after completing all substrate tests (no weighing).

4.0 DEVIATIONS FROM PROTOCOL

Every attempt shall be made to follow this protocol. Deviations from the protocol may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual or unanticipated conditions.

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the team leader and the MRI Work Assignment Leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation shall be recollected in accordance with the specified protocol.

Deliberate deviations from the sampling protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be expedited. The MRI Work Assignment Leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals.

Test Sequence for Fiber Preconditioning of Carpets and Upholstery

Table shows vacuumings within tests

Cycle through the vacuums until two successive vacuums collect less than 20 mg. of dust per 5 minutes of vacuuming
The order of the substrates and the choice of teams is not important
Put a new bag in each vacuum each morning

Test number	Substrate sample: Substrate	Dust Loading	Lead Conc	Particle size	New bag	Dust deposited (mg)	Vacuum	Team	time vacuumed	weight increase (mg)	dust sent for analysis (mg)	lead amount (ug)	Date Vacuumed	Time vacuumed
1.01	Carpet	400 mg/sq ft	High		no?		A		5 min					
1.02	"	"	"		no?		B		5 min					
1.03	"	"	"		no?		C		5 min					
1.04	"	"	"		no?		D		5 min					
1.05	"	"	"		no?		...		...					
....	"	"	"		no?									
2.01	Carpet	400 mg/sq ft	Low		no?		B		5 min					
2.02	"	"	"		no?		C		5 min					
2.03	"	"	"		no?		D		5 min					
2.04	"	"	"		no?		A		5 min					
2.05	"	"	"		no?		...		...					
....	"	"	"		no?									
3.01	Carpet	100 mg/sq ft	Low		no?		C		5 min					
3.02	"	"	"		no?		D		5 min					
3.03	"	"	"		no?		A		5 min					
3.04	"	"	"		no?		B		5 min					
3.05	"	"	"		no?		...		...					
....	"	"	"		no?									
4.01	Carpet	100 mg/sq ft	High		no?		D		5 min					
4.02	"	"	"		no?		A		5 min					
4.03	"	"	"		no?		B		5 min					
4.04	"	"	"		no?		C		5 min					
4.05	"	"	"		no?		...		...					
....	"	"	"		no?									
5.01	Carpet with Grind-in	100 mg/sq ft	Low		no?		B		5 min					
5.02	"	"	"		no?		C		5 min					
5.03	"	"	"		no?		D		5 min					
5.04	"	"	"		no?		A		5 min					
5.05	"	"	"		no?		...		...					
....	"	"	"		no?									
6.01	Carpet with Grind-in	100 mg/sq ft	High		no?		C		5 min					
6.02	"	"	"		no?		D		5 min					
6.03	"	"	"		no?		A		5 min					
6.04	"	"	"		no?		B		5 min					
6.05	"	"	"		no?		...		...					
....	"	"	"		no?									

Test Sequence for Fiber Preconditioning of Carpets and Upholstery (continued)

Fiber preconditioning (continued)
Table shows vacuumings within tests

Test number	Substrate sample: Substrate	Dust Loading	Lead Conc	Particle size	New bag	Dust deposited (mg)	Vacuum	Team	time vacuumed	weight increase (mg)	dust sent for analysis (mg)	lead amount (ug)	Date Vacuumed	Time vacuumed
7.01	Carpet with Grind-in	400 mg/sq ft	Low		no?		D		5 min					
7.02	"	"	"		no?		A		5 min					
7.03	"	"	"		no?		B		5 min					
7.04	"	"	"		no?		C		5 min					
7.05	"	"	"		no?		...		...					
....	"	"	"		no?									
8.01	Carpet with Grind-in	400 mg/sq ft	High		no?		A		5 min					
8.02	"	"	"		no?		B		5 min					
8.03	"	"	"		no?		C		5 min					
8.04	"	"	"		no?		D		5 min					
8.05	"	"	"		no?		...		...					
....	"	"	"		no?									
9.01	Upholstery	100 mg/sq ft	Low		no?		C		5 min					
9.02	"	"	"		no?		D		5 min					
9.03	"	"	"		no?		A		5 min					
9.04	"	"	"		no?		B		5 min					
9.05	"	"	"		no?		...		...					
....	"	"	"		no?									
10.01	Upholstery	100 mg/sq ft	High		no?		D		5 min					
10.02	"	"	"		no?		A		5 min					
10.03	"	"	"		no?		B		5 min					
10.04	"	"	"		no?		C		5 min					
10.05	"	"	"		no?		...		...					
....	"	"	"		no?									
11.01	Upholstery	400 mg/sq ft	High		no?		A		5 min					
11.02	"	"	"		no?		B		5 min					
11.03	"	"	"		no?		C		5 min					
11.04	"	"	"		no?		D		5 min					
11.05	"	"	"		no?		...		...					
....	"	"	"		no?									
12.01	Upholstery	400 mg/sq ft	Low		no?		B		5 min					
12.02	"	"	"		no?		C		5 min					
12.03	"	"	"		no?		D		5 min					
12.04	"	"	"		no?		A		5 min					
12.05	"	"	"		no?		...		...					
....	"	"	"		no?									

**Data Entry Sheet
for
Fiber Preconditioning:
First Vacuum—A**

Test Identification

Test number ____________
Date ____________
Operator ____________
Time ____________

Substrate ____________ (upholstery, carpet)

Substrate to be used for:
- Grind-in ________ (Yes or No)
- Dust Amount ________ (100, 400 mg/sq ft)
- Pb Conc ________ (Low, High)

Procedure

Use a new bag for each vac each morning
Tare weigh each new bag prior to first use:
Run free for 5 min, cool 2 min, brush, place in plastic bag and record weight after 1 more min
Vac carpet for 5 min, starting with vac identified in above heading
Reweigh after vac carpet for 5 min (cool 2 min, brush, place back in plastic bag and record weight after 1 more min)
Proceed with 5 min vacuumings with each vac. For tare weight use previous final weight from other sheet
Continue 5 min vacuumings until weight change is <20 mg for 4 vacs consecutively, or <40 mg for 8 vacs consecutively

	A (Balance No. ______)			B (Balance No. ______)			C (Balance No. ______)			D (Balance No. ______)		
	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm
01	____	____	____									
.02				____	____	____						
.03							____	____	____			
.04										____	____	____
.05	____	____	____									
.06				____	____	____						
.07							____	____	____			
.08										____	____	____
.09	____	____	____									
.10				____	____	____						
.11							____	____	____			
.12										____	____	____
.13	____	____	____									
.14				____	____	____						
.15							____	____	____			
.16										____	____	____
.17	____	____	____									
.18				____	____	____						
.19							____	____	____			
.20										____	____	____

Reviewed by ____________
Date reviewed ____________

Data Entry Sheet
for
Fiber Preconditioning:
First Vacuum—B

Test Identification

Test number ______________
Date ______________
Operator ______________
Time ______________

Substrate ______________ (upholstery, carpet)

Substrate to be used for:
- Grind-in __________ (Yes or No)
- Dust Amount __________ (100, 400 mg/sq ft)
- Pb Conc __________ (Low, High)

Procedure

Use a new bag for each vac each morning
Tare weigh each new bag prior to first use:
Run free for 5 min, cool 2 min, brush, place in plastic bag and record weight after 1 more min
Vac carpet for 5 min, starting with vac identified in above heading
Reweigh after vac carpet for 5 min (cool 2 min, brush, place back in plastic bag and record weight after 1 more min)
Proceed with 5 min vacuumings with each vac. For tare weight use previous final weight from other sheet
Continue 5 min vacuumings until weight change is <20 mg for 4 vacs consecutively, or <40 mg for 8 vacs consecutively

	B (Balance No. ________)			C (Balance No. ________)			D (Balance No. ________)			A (Balance No. ________)		
	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm
01	____	____	____									
.02				____	____	____						
.03							____	____	____			
.04										____	____	____
.05	____	____	____									
.06				____	____	____						
.07							____	____	____			
.08										____	____	____
.09	____	____	____									
.10				____	____	____						
.11							____	____	____			
.12										____	____	____
.13	____	____	____									
.14				____	____	____						
.15							____	____	____			
.16										____	____	____
.17	____	____	____									
.18				____	____	____						
.19							____	____	____			
.20										____	____	____

Reviewed by ______________
Date reviewed ______________

Data Entry Sheet
for
Fiber Preconditioning:
First Vacuum—C

Test Identification

Substrate ______________ (upholstery, carpet)

Test number ______________
Date ______________
Operator ______________

Time ______________

Substrate to be used for:
- Grind-in __________ (Yes or No)
- Dust Amount __________ (100, 400 mg/sq ft)
- Pb Conc __________ (Low, High)

Procedure

Use a new bag for each vac each morning
Tare weigh each new bag prior to first use:
Run free for 5 min, cool 2 min, brush, place in plastic bag and record weight after 1 more min
Vac carpet for 5 min, starting with vac identified in above heading
Reweigh after vac carpet for 5 min (cool 2 min, brush, place back in plastic bag and record weight after 1 more min)
Proceed with 5 min vacuumings with each vac. For tare weight use previous final weight from other sheet
Continue 5 min vacuumings until weight change is <20 mg for 4 vacs consecutively, or <40 mg for 8 vacs consecutively

	C (Balance No. ________)			D (Balance No. ________)			A (Balance No. ________)			B (Balance No. ________)		
	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm
01	____	____	____									
.02				____	____	____						
.03							____	____	____			
.04										____	____	____
.05	____	____	____									
.06				____	____	____						
.07							____	____	____			
.08										____	____	____
.09	____	____	____									
.10				____	____	____						
.11							____	____	____			
.12										____	____	____
.13	____	____	____									
.14				____	____	____						
.15							____	____	____			
.16										____	____	____
.17	____	____	____									
.18				____	____	____						
.19							____	____	____			
.20										____	____	____

Reviewed by ______________
Date reviewed ______________

Data Entry Sheet
for
Fiber Preconditioning:
First Vacuum—D

Test Identification

Test number ____________
Date ____________
Operator ____________

Time ____________

Substrate ____________ (upholstery, carpet)

Substrate to be used for:
- Grind-in ____________ (Yes or No)
- Dust Amount ____________ (100, 400 mg/sq ft)
- Pb Conc ____________ (Low, High)

Procedure

Use a new bag for each vac each morning
Tare weigh each new bag prior to first use:
Run free for 5 min, cool 2 min, brush, place in plastic bag and record weight after 1 more min
Vac carpet for 5 min, starting with vac identified in above heading
Reweigh after vac carpet for 5 min (cool 2 min, brush, place back in plastic bag and record weight after 1 more min)
Proceed with 5 min vacuumings with each vac. For tare weight use previous final weight from other sheet
Continue 5 min vacuumings until weight change is <20 mg for 4 vacs consecutively, or <40 mg for 8 vacs consecutively

	D (Balance No. ______)			A (Balance No. ______)			B (Balance No. ______)			C (Balance No. ______)		
	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm	Final Wt. gm	Tare Wt gm	Weight Chg gm
01	____	____	____									
.02				____	____	____						
.03							____	____	____			
.04										____	____	____
.05	____	____	____									
.06				____	____	____						
.07							____	____	____			
.08										____	____	____
.09	____	____	____									
.10				____	____	____						
.11							____	____	____			
.12										____	____	____
.13	____	____	____									
.14				____	____	____						
.15							____	____	____			
.16										____	____	____
.17	____	____	____									
.18				____	____	____						
.19							____	____	____			
.20										____	____	____

Reviewed by ____________
Date reviewed ____________

Test Sequence for Dust Preconditioning (Team 1)

Dust preconditioning, Team 1
Table shows sequence of tests by team

Test number	Substrate sample: Substrate	Dust Loading	Lead Conc	Particle size	New bag	Dust deposited (mg)	Vacuum	Team	time vacuumed	dust collected (mg)	dust sent for analysis (mg)	lead amount (ug)	Date Vacuumed	Time vacuumed	Target Dust deposited (g)
101	Carpet	400 mg/sq ft	Low	150-212	n		D	1	40 sec						2.712
102	Carpet	400 mg/sq ft	Low	250-2000	n		B	1	40 sec						2.712
103	Carpet	400 mg/sq ft	Low	53-106	n		B	1	40 sec						2.712
104	Carpet	400 mg/sq ft	Low	106-150	n		C	1	40 sec						2.712
105	Carpet	400 mg/sq ft	Low	212-250	n		A	1	40 sec						2.712
106	Carpet	400 mg/sq ft	Low	<53	n		C	1	40 sec						2.712
107	Upholstery	400 mg/sq ft	Low	106-150	n		A	1	40 sec						2.712
108	Upholstery	400 mg/sq ft	Low	<53	n		B	1	40 sec						2.712
109	Upholstery	400 mg/sq ft	Low	250-2000	n		D	1	40 sec						2.712
110	Upholstery	400 mg/sq ft	Low	212-250	n		C	1	40 sec						2.712
111	Upholstery	400 mg/sq ft	Low	150-212	n		A	1	40 sec						2.712
112	Upholstery	400 mg/sq ft	Low	53-106	n		D	1	40 sec						2.712
113	Upholstery	100 mg/sq ft	High	106-150	n		D	1	40 sec						0.678
114	Upholstery	100 mg/sq ft	High	<53	n		D	1	40 sec						0.678
115	Upholstery	100 mg/sq ft	High	150-212	n		A	1	40 sec						0.678
116	Upholstery	100 mg/sq ft	High	212-250	n		B	1	40 sec						0.678
117	Upholstery	100 mg/sq ft	High	53-106	n		C	1	40 sec						0.678
118	Upholstery	100 mg/sq ft	High	250-2000	n		C	1	40 sec						0.678
119	Carpet	100 mg/sq ft	High	150-212	n		C	1	40 sec						0.678
120	Carpet	100 mg/sq ft	High	106-150	n		D	1	40 sec						0.678
121	Carpet	100 mg/sq ft	High	53-106	n		A	1	40 sec						0.678
122	Carpet	100 mg/sq ft	High	250-2000	n		A	1	40 sec						0.678
123	Carpet	100 mg/sq ft	High	<53	n		B	1	40 sec						0.678
124	Carpet	100 mg/sq ft	High	212-250	n		D	1	40 sec						0.678
125	Carpet with Grind-in	100 mg/sq ft	Low	<53	n		D	1	40 sec						0.678
126	Carpet with Grind-in	100 mg/sq ft	Low	106-150	n		B	1	40 sec						0.678
127	Carpet with Grind-in	100 mg/sq ft	Low	250-2000	n		A	1	40 sec						0.678
128	Carpet with Grind-in	100 mg/sq ft	Low	212-250	n		D	1	40 sec						0.678
129	Carpet with Grind-in	100 mg/sq ft	Low	150-212	n		C	1	40 sec						0.678
130	Carpet with Grind-in	100 mg/sq ft	Low	53-106	n		A	1	40 sec						0.678
131	aSheet vinyl	400 mg/sq ft	Low	106-150	n		B	1	40 sec						2.712
132	aSheet vinyl	100 mg/sq ft	Low	106-150	n		D	1	40 sec						0.678
133	aSheet vinyl	100 mg/sq ft	High	106-150	n		A	1	40 sec						0.678
134	aSheet vinyl	400 mg/sq ft	High	106-150	n		C	1	40 sec						2.712
135	aWood	100 mg/sq ft	Low	106-150	n		B	1	40 sec						0.678
136	aWood	100 mg/sq ft	High	106-150	n		C	1	40 sec						0.678
137	Carpet with Grind-in	400 mg/sq ft	High	150-212	n		B	1	40 sec						2.712
138	Carpet with Grind-in	400 mg/sq ft	High	106-150	n		A	1	40 sec						2.712
139	Carpet with Grind-in	400 mg/sq ft	High	212-250	n		C	1	40 sec						2.712
140	Carpet with Grind-in	400 mg/sq ft	High	53-106	n		D	1	40 sec						2.712
141	Carpet with Grind-in	400 mg/sq ft	High	<53	n		A	1	40 sec						2.712
142	Carpet with Grind-in	400 mg/sq ft	High	250-2000	n		D	1	40 sec						2.712

Dust preconditioning, Team 2
Table shows sequence of tests by team

Test Sequence for Dust Preconditioning (Team 2)

Test number	Substrate sample: Substrate	Substrate sample: Dust Loading	Substrate sample: Lead Conc	Particle size	New bag	Dust deposited (mg)	Vacuum	Team	time vacuumed	dust collected (mg)	dust sent for analysis (mg)	lead amount (ug)	Date Vacuumed	Time vacuumed	Target Dust deposited (g)
201	Tile	100 mg/sq ft	Low	106-150	n		A	2	40 sec						0.678
202	Tile	400 mg/sq ft	High	106-150	n		D	2	40 sec						2.712
203	Tile	400 mg/sq ft	Low	106-150	n		C	2	40 sec						2.712
204	Tile	100 mg/sq ft	High	106-150	n		B	2	40 sec						0.678
205	Wood	400 mg/sq ft	Low	106-150	n		D	2	40 sec						2.712
206	Wood	400 mg/sq ft	High	106-150	n		A	2	40 sec						2.712
207	Carpet	400 mg/sq ft	High	212-250	n		B	2	40 sec						2.712
208	Carpet	400 mg/sq ft	High	250-2000	n		C	2	40 sec						2.712
209	Carpet	400 mg/sq ft	High	53-106	n		C	2	40 sec						2.712
210	Carpet	400 mg/sq ft	High	106-150	n		B	2	40 sec						2.712
211	Carpet	400 mg/sq ft	High	<53	n		D	2	40 sec						2.712
212	Carpet	400 mg/sq ft	High	150-212	n		A	2	40 sec						2.712
213	Carpet	100 mg/sq ft	Low	212-250	n		C	2	40 sec						0.678
214	Carpet	100 mg/sq ft	Low	106-150	n		A	2	40 sec						0.678
215	Carpet	100 mg/sq ft	Low	53-106	n		D	2	40 sec						0.678
216	Carpet	100 mg/sq ft	Low	250-2000	n		D	2	40 sec						0.678
217	Carpet	100 mg/sq ft	Low	<53	n		A	2	40 sec						0.678
218	Carpet	100 mg/sq ft	Low	150-212	n		B	2	40 sec						0.678
219	Carpet with Grind-in	400 mg/sq ft	Low	106-150	n		D	2	40 sec						2.712
220	Carpet with Grind-in	400 mg/sq ft	Low	<53	n		A	2	40 sec						2.712
221	Carpet with Grind-in	400 mg/sq ft	Low	250-2000	n		C	2	40 sec						2.712
222	Carpet with Grind-in	400 mg/sq ft	Low	150-212	n		B	2	40 sec						2.712
223	Carpet with Grind-in	400 mg/sq ft	Low	53-106	n		C	2	40 sec						2.712
224	Carpet with Grind-in	400 mg/sq ft	Low	212-250	n		B	2	40 sec						2.712
225	Upholstery	100 mg/sq ft	Low	250-2000	n		B	2	40 sec						0.678
226	Upholstery	100 mg/sq ft	Low	106-150	n		C	2	40 sec						0.678
227	Upholstery	100 mg/sq ft	Low	150-212	n		D	2	40 sec						0.678
228	Upholstery	100 mg/sq ft	Low	53-106	n		B	2	40 sec						0.678
229	Upholstery	100 mg/sq ft	Low	212-250	n		A	2	40 sec						0.678
230	Upholstery	100 mg/sq ft	Low	<53	n		C	2	40 sec						0.678
231	Carpet with Grind-in	100 mg/sq ft	High	<53	n		C	2	40 sec						0.678
232	Carpet with Grind-in	100 mg/sq ft	High	106-150	n		C	2	40 sec						0.678
233	Carpet with Grind-in	100 mg/sq ft	High	53-106	n		B	2	40 sec						0.678
234	Carpet with Grind-in	100 mg/sq ft	High	212-250	n		A	2	40 sec						0.678
235	Carpet with Grind-in	100 mg/sq ft	High	150-212	n		D	2	40 sec						0.678
236	Carpet with Grind-in	100 mg/sq ft	High	250-2000	n		B	2	40 sec						0.678
237	Upholstery	400 mg/sq ft	High	212-250	n		D	2	40 sec						2.712
238	Upholstery	400 mg/sq ft	High	106-150	n		B	2	40 sec						2.712
239	Upholstery	400 mg/sq ft	High	250-2000	n		A	2	40 sec						2.712
240	Upholstery	400 mg/sq ft	High	53-106	n		A	2	40 sec						2.712
241	Upholstery	400 mg/sq ft	High	150-212	n		C	2	40 sec						2.712
242	Upholstery	400 mg/sq ft	High	<53	n		B	2	40 sec						2.712

Dust preconditioning will require 21 grams of dust for each particle size and lead level, except 42 grams of size 106-150 um

**Dust Entry Sheets
For
Dust Preconditioning**

Test Sequence Numbers ____________
Data ____________
Operator ____________

Test Identification

Substrate	________	(TILE, LINOleum, WOOD, UPHOLstery, CaPReT)
Grind-in	________	(Yes, No)
Dust Amount	________	(100, 400 mg/sq ft)
Pb Conc	________	(Low, High)
Dust Size	(see below)	
Team	________	(number 1 or 2)

Procedure

Use a new bag in each vacuum at the beginning of the day
Perform the tests according to the test sequence for dust preconditioning
Deposit dust. Determine the actual weight of dust deposited
Grind-in if applicable
Determine tare weight of bag before each use:
Run free for 40 sec, cool 2 min, brush and record weight after 1 more min
Vacuum for 40 sec with the vacuum indicated in the test sequence for dust preconditioning
Record the time of the vacuuming
Reweigh bag after 40 sec vac (cool 2 min, brush and record weight after 1 more min)
Repeat tare-vac-reweigh using the housevac and particle size designated in test sequence, which utilizes the same substrate with the same dust loading and lead conc.
Vacuum the wand and brush all housevacs after completing all tests on the substrate (no weighing)

			Weight of Dust Applied (g) (Balance No.________)			Weight of Bag (g) (Balance No. ________)		
Test Number (Time)	House Vac	Particle Size	Total Wt	Tare Wt	Net Wt	Final Wt	Tare Wt	Net Wt
( )								
( )								
( )								
( )								
( )								
( )								

APPENDIX E

PROTOCOL FOR WIPE SAMPLING OF DUST

PROTOCOL FOR WIPE SAMPLING OF DUST

1.0 INTRODUCTION

Wipe samples of dust will be collected from substrates (except carpet and upholstery) using commercially available moistened disposable wipes (Wash-a-bye Baby® brand). The surfaces will be wiped using a sampling method developed by Dr. Farfel for his doctoral thesis at Johns Hopkins University, School of Hygiene and Public Health (Farfel, 1987). This sampling method is also found in the National Institute of Building Sciences "Guidelines for Testing, Abatement, Clean Up, and Disposal of Lead-Based Paint in Housing."

2.0 SAMPLING EQUIPMENT AND SUPPLIES

The following materials will be used to collect wipe samples:

- Wash-a-bye Baby® wipes
- Washable template (inside dimensions, 1 ft by 1 ft)
- Steel measuring tape
- Marking pen
- 1-quart plastic bags
- Disposable vinyl gloves (powderless)
- Substrates (tile, linoleum, wood)

3.0 WIPE SAMPLING PROTOCOL

The following procedure will be used to wipe sample substrate surfaces:

- Don a pair of clean, powderless, vinyl gloves.

- Remove the seal on a package containing the wipes (if not already removed during previous sampling efforts), open the lid, start the lid dispenser, replace the lid, remove several wipes, and discard them in the black trash bag. Use the next wipe from the container to collect the sample.

- Position a clean 1-ft^2 template on the surface to be sampled.

- Place the wipe flat on the surface within the sample area as defined by the template. Using an open flat hand with the fingers together wipe the marked surface in an overlapping "S" pattern, first side to side and then front to back so that the entire 1-ft^2 area is covered.

- Fold the wipe in half with the sample side folded in and repeat the wiping procedure within the marked surface area on one side of the folded wipe.

- Fold the wipe again with the sample side folded in.

- Insert the folded wipe into the inner ziplock plastic bag and seal. Attach barcode label to the bag and a duplicate barcode label on the sampling form. Place the plastic bag inside another plastic bag along with extra duplicate barcode labels.

- Remove the vinyl gloves and dispose of them in the black trash bag.

- Record test number, date, time, etc., on the sampling data form.

4.0 PREPARATION OF SAMPLING BLANK SAMPLE

The sampling blank will consist of a Wash-a-bye Baby™ wipe that is handled using the identical procedure used for the dust samples except that no sample is collected. Provide a blank sample with each batch of samples, or once each week as a minimum.

5.0 CONTAMINATION AVOIDANCE

The following work practices will be instituted to prevent cross-contamination between samples collected:

- Clean vinyl gloves (powderless) will be donned prior to collecting each wipe sample and will be disposed of after each sample is collected.
- The templates will be cleaned with a Wash-a-bye Baby® disposable wet wipe between each use. After cleaning the template, remove the vinyl gloves and dispose of them.

6.0 DEVIATIONS FROM THE WIPE SAMPLING PROTOCOL

Every attempt shall be made to follow this sampling protocol. Deviations from the sampling protocols may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual conditions encountered in the laboratory).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the MRI work assignment leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation should be recollected in accordance with the specified protocol prior to leaving the site.

Deliberate deviations from the sampling protocol must be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA and MRI. These verbal approvals will be followed by a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can by expedited. The MRI work assignment leader is responsible for initiation of the QAPjP modification and for acquiring the necessary approvals from EPA and MRI.

The work assignment leader shall be notified by the sampling team when conditions found in the laboratory do not allow full compliance with the protocol or when the protocol does not appear to apply to the situation. The condition/situation shall be fully documented in a laboratory notebook.

Data Entry Sheets
for
Sampler Tests

Test Sequence Number ____________
Date ____________
Operator ____________

Test Identification

Sampler	_____	(Blue Nozzle, CAPS, HVS3 or WIPE)
Substrate	_____	(TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in	_____	(Yes, No)
Dust Amount	_____	(100, 400 mg/ft^2)
Pb Conc	_____	(Low, High)
Dust Size	_____	(>53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team	_____	(Number 1 or 2)
Square number	_____	(1, 2, 3 or 4) 1=first, 3=last for carpet and upholstery, else 4=last)

Procedure

Perform the tests according to the sampler test sequence in Appendix Q, and procedures in Appendix E, F, G or H. Housevac A will be used to vacuum the first square before sampler tests, or to vacuum the last square after sampler tests.

If first square:

- Tare weigh bag (run free for 40 seconds, cool 2 minutes, brush and record weight after 1 more minute)
- Vac square for 40 seconds with Housevac A
- Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)

Deposit dust in specified square and weigh the amount deposited (Grind-in dust if applicable)
Sample dust according to the appropriate protocol, weigh the dust collected (except for wipes)
Prepare the dust sample for analysis
If last square:

- Tare weigh bag (run free for 120 seconds, cool 2 minutes, brush and record weight after 1 more minute)
- Vac square for 120 seconds with Housevac A
- Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)

Vacuum dust from wand and brush (no weighing)

		Weight of Dust (Balance #_______)				Weight of Bag (Balance #_______)	
		Total Wt. gm.	Final Wt. gm.	Net Wt. gm.	Time	Weight gm.	Increase gm.
Initial weight of bag (if first or last square)	.0					_____	
Vacuum and reweigh bag (if first square)	.1					_____	_____
Dust deposited	.2	_____	_____	_____	_____		
Dust collected by sampler (exclu wipes)	.3	_____	_____	_____			
Vacuum & reweigh bag (if last square)	4.					_____	_____

Bar Code for Sample	Bar Code for Blank

NOTE: Submit one blank for each sampler, once each week

Sample relinquished by ____________ Reviewed by ____________
Sample received by ____________ Date reviewed ____________
Date of transfer ____________

APPENDIX F

PROTOCOL FOR VACUUM SAMPLING OF DUST WITH THE BRM CYCLONE SAMPLER VAC

PROTOCOL FOR VACUUM SAMPLING OF DUST WITH THE BRM CYCLONE SAMPLER VAC

1.0 INTRODUCTION

Vacuum samples of dust will be collected from floors (carpeted and uncarpeted) and upholstery material as specified by the QAPjP. The vacuum sampling device is a BRM cyclone dust collector shown in Figure F-1.

Each 1-ft^2 section of the surface to be sampled will be vacuumed in overlapping passes (Figure F-2). A 1-ft^2 template will be used to define and measure the areas to be vacuumed.

2.0 SAMPLING EQUIPMENT AND SUPPLIES

- Modified BRM cyclone dust collectors
- 12-in Remote switch
- Vinyl tubing (³/₄-in ID)
- Dust catch containers (for the modified BRM cyclone)
- Plastic bottles for collection of dust samples
- 1-ft^2 templates (full square)
- Stainless steel dust collector
- Steel measuring tape
- Screwdriver (tapping cyclone dust samples, if necessary)

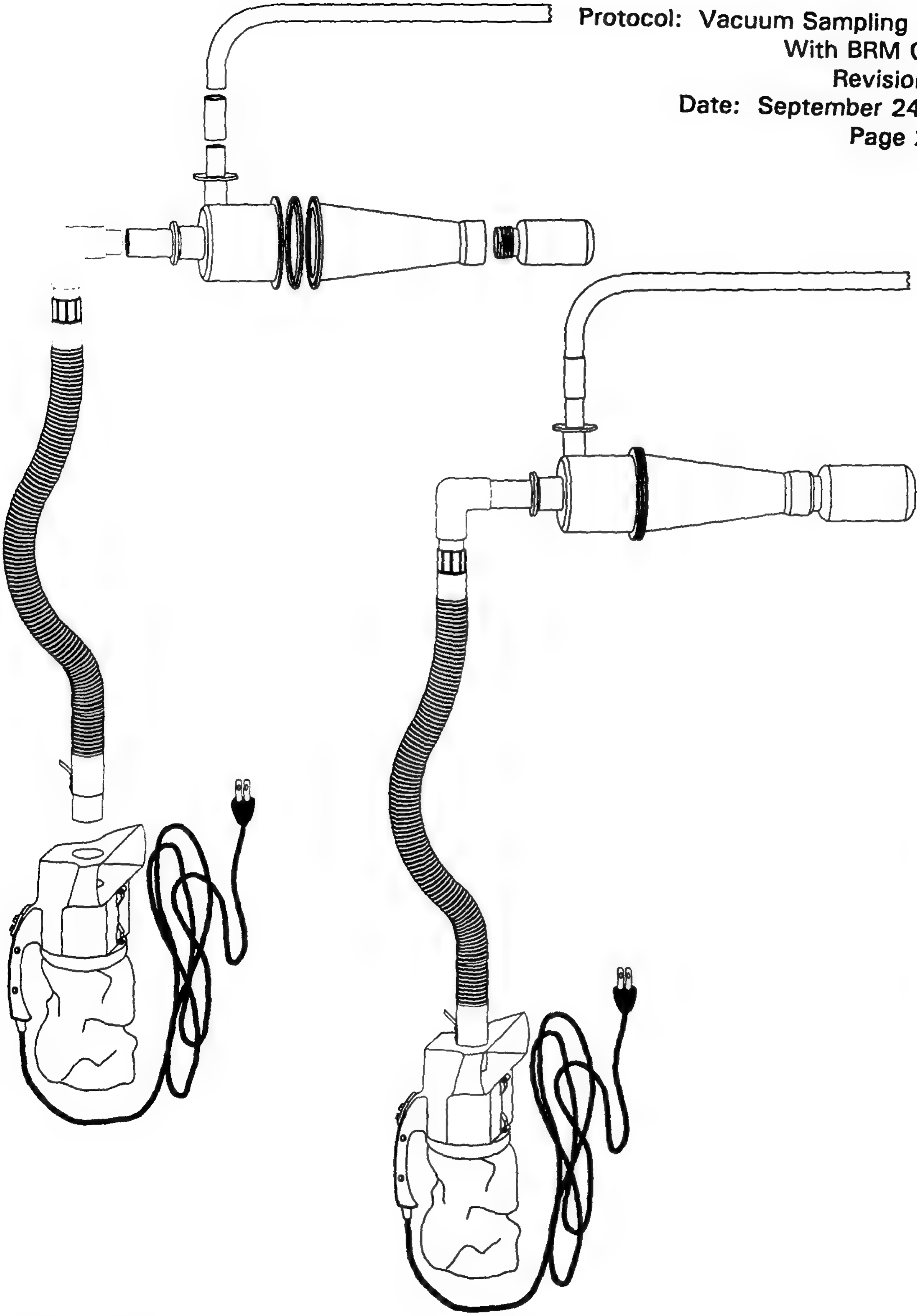

Figure F-1. BRM cyclone dust collector.

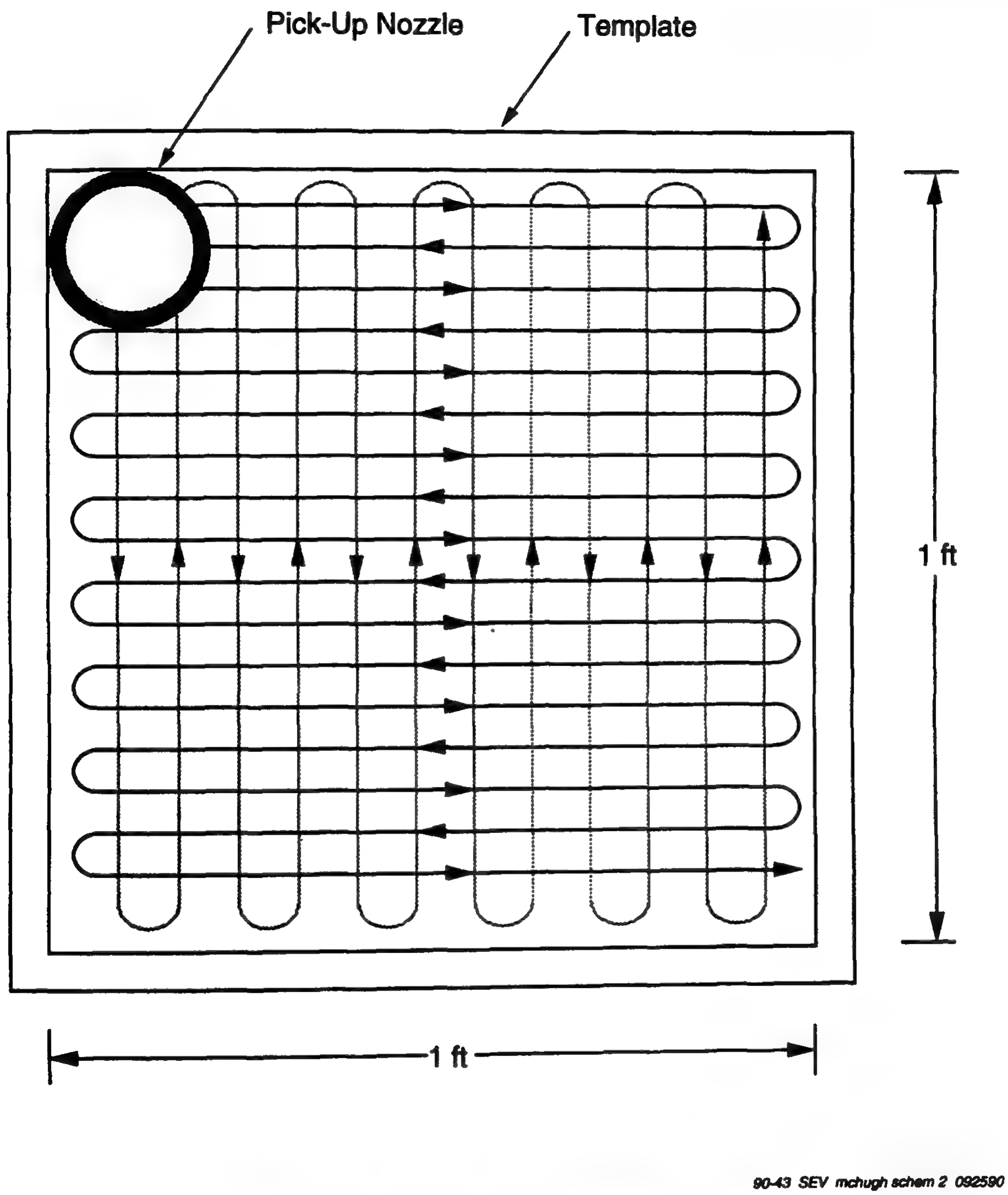

Figure F-2. Vacuuming sampling pattern.

- Tweezers (long 12-in).
- Timing device (stopwatch, timer, or watch with second hand).
- Barcode labels (12 identical labels per sample with a unique sample number).
- 1-qt and 1-gal ziplock plastic bags
- Sample data sheets
- Vinyl gloves (powderless)
- Wash-a-bye Baby® premoistened disposable wipes to clean equipment
- Large Kimwipes®

3.0 VACUUM SAMPLING PROTOCOL

The following protocol will be used to collect vacuum samples of dust.

3.1 Sampling Preparations

- Use a clean 1-ft^2 template to define the surface area to be vacuumed.
- Record test number, date, time, etc., on the sampling data form.

3.2 Cyclone Vacuum Assembly

- The cyclone vacuum consists of four major parts (see Figure F-3).
    - Head
    - Case
    - Teflon ring
    - Clamp

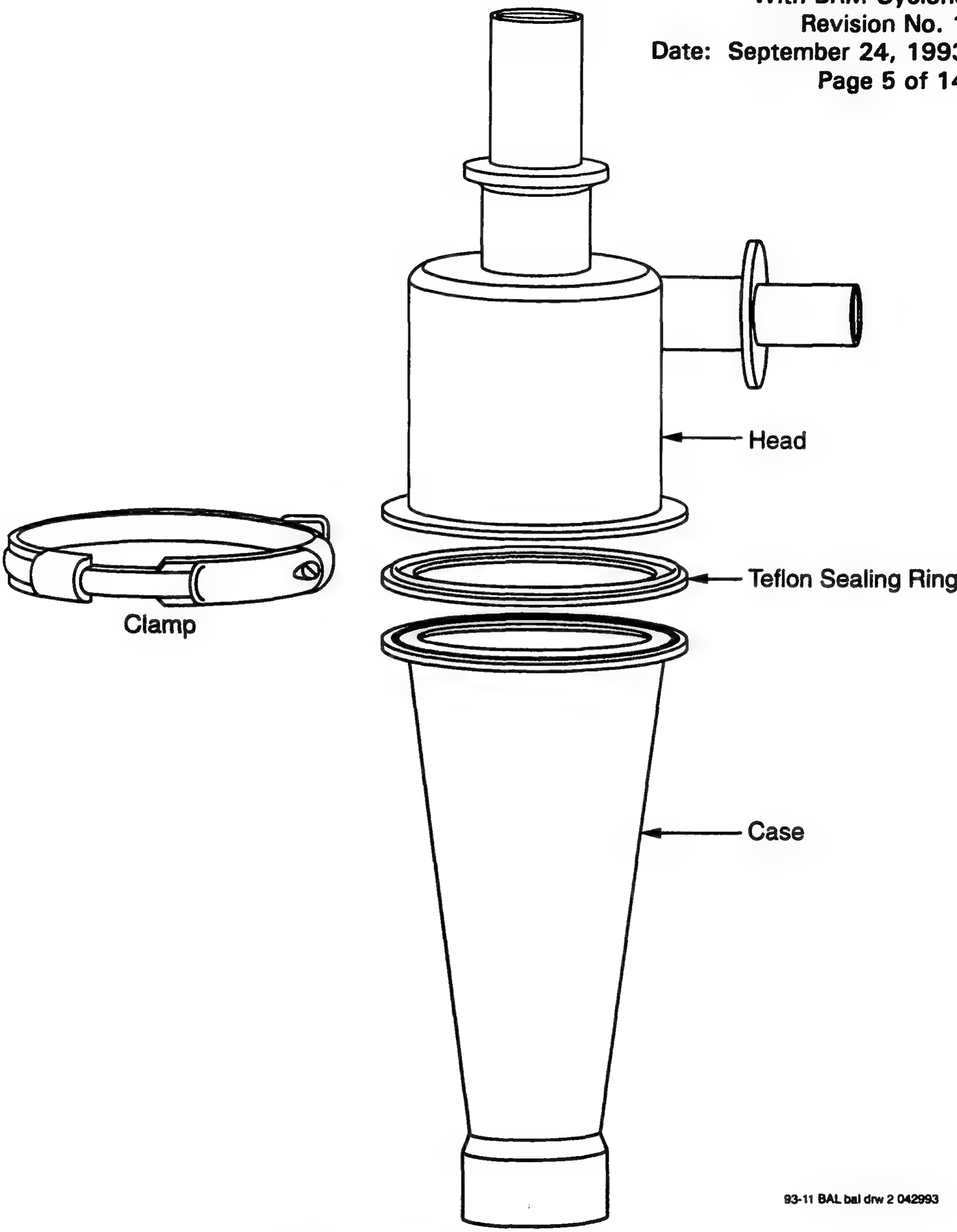

Figure F-3. BRM cyclone vacuum assembly.

The head of the cyclone has an output nozzle located on the top that would be connected to a vacuum source and an inlet located tangentially on the side that would be connected to a pick-up tube.

The conical shaped bottom half of the cyclone vacuum is the case. Internal threads are located at the narrow end of the case. The dust catch container will be screwed tightly into these threads.

A Teflon sealing ring is located between the head and case of the cyclone. The clamp holds the head and case together.

3.3 Cyclone Dust Collector Preparation

- Properly support the cyclone dust collector for disassembly.
- Disengage the clamping device used to hold the head and case together.
- Remove the clamp and separate the head and case.
- Wipe the inside surfaces of the cyclone's head and case with a Wash-A-Bye Baby® wipe. Long tweezers or other devices may be required to reach all areas to wipe. Use more than one wipe if necessary.
- Rewipe the inside surfaces of the cyclone head and case with a clean, dry Kimwipe®.
- Place the used wipes in a waste container.
- Reassemble cyclone head and case. Be sure Teflon sealing ring is in its proper position (Figure F-3). Before engaging clamp, make sure head and case are lined up correctly. If the clamp appears to be more difficult than normal to engage, check for misalignment.
- Affix the hand vacuum to the cyclone sampler case as shown in Figure F-1.
- Don a pair of powderless vinyl gloves prior to handling the sampling dust catch container.

- Obtain a sampling dust container (plastic bottle with lid). Affix a barcode label to the container and an identical barcode label on the associated sampling data form.

- Weigh the sample container (with label and lid) and record weighings on test form. Weigh the container twice or as many times as necessary until two weighings agree within ±2 mg.

- Place a clean grey PVC transition piece onto the tangential inlet of the cyclone head. This piece has two different IDs. The proper end of the transition piece should fit snugly onto the inlet tube.

- Position the cyclone in a vertical position.

- Securely screw the sampling container into the lower threaded end of the cyclone case (see Figure F-4).

- Insert the proper end of the pick-up tube into the grey transition piece mentioned above.

- Attach the vinyl tubing (Figure F-5) to the sampler case's 3/4-in inlet.

3.4 Vacuuming Procedures for the BRM Cyclone

- Plug the hand vacuum in a 110-V AC outlet.

  NOTE: The vacuum unit comes with a 12-ft vacuum extension hose and a remote switch. This allows the vacuum unit to be located away from the sampling area while on/off control is maintained at the sampling location.

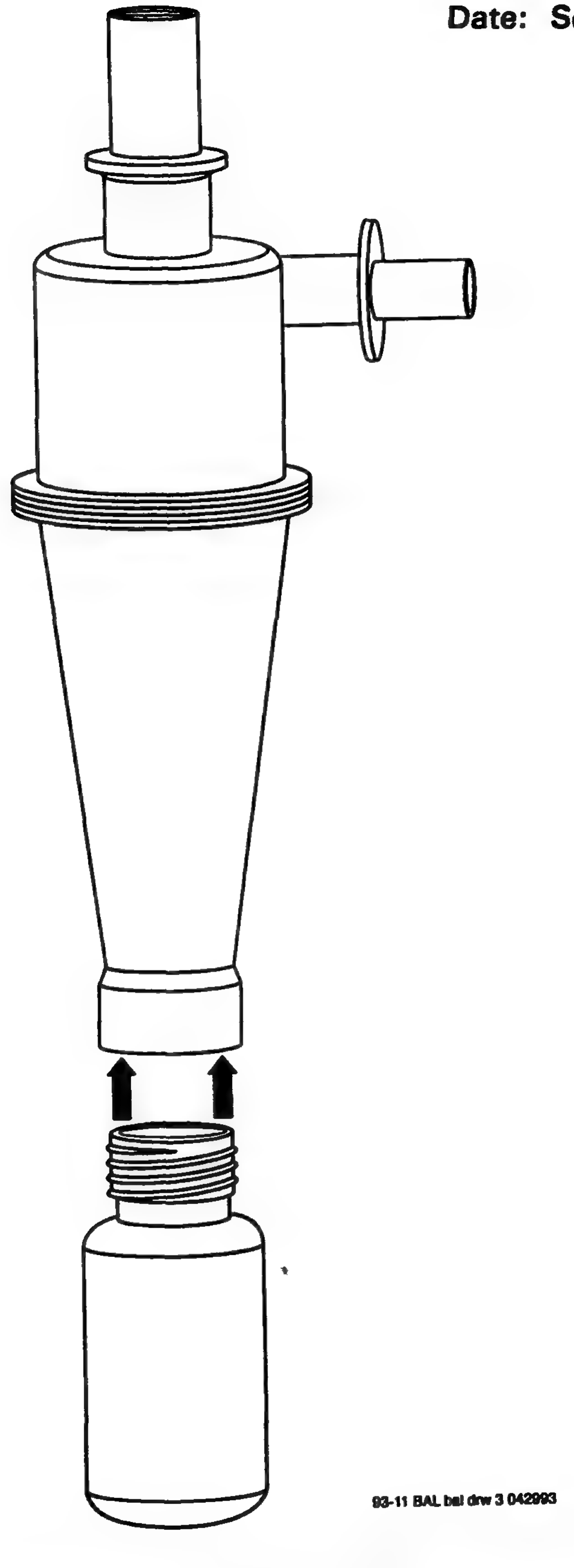

Figure F-4. Sample collection bottle with cyclone assembly.

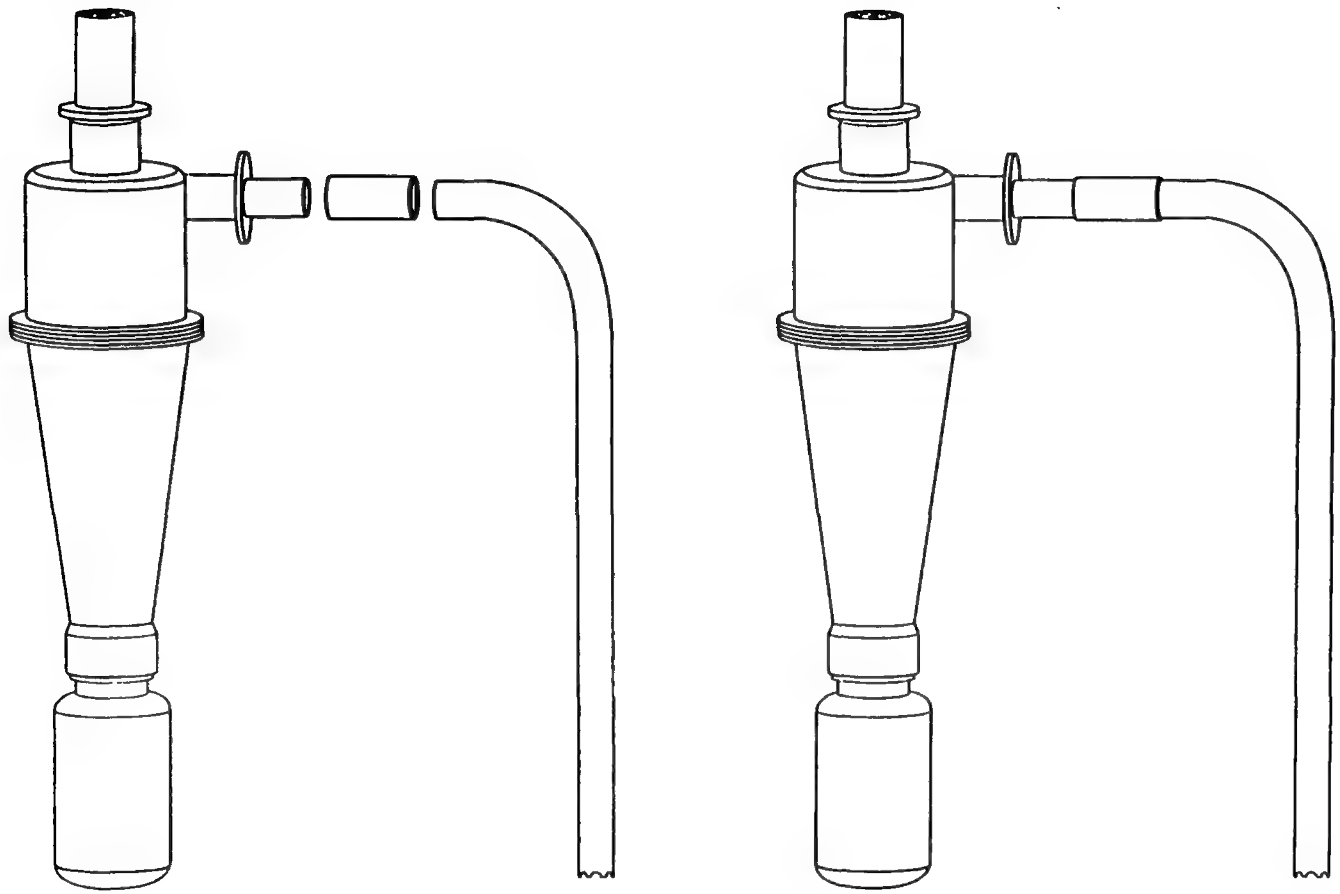

Figure F-5. Attachment of vinyl tubing.

- Turn on the pump and vacuum the area of interest evenly in overlapping passes (at least 50% overlap), first left to right, then front to back over the entire designated area (Figure F-2). Vacuum the area again using this same pattern. For a 1-ft^2 area, vacuuming should not exceed 2 min.

  NOTE: The cyclone sampler case must be held vertically throughout the vacuuming process through the removal of the sampling container.

  NOTE: The template used to define a surface area to be vacuumed is the potential source of cross-contamination between samples. The template must be thoroughly cleaned with disposable wipes between each sample.

- When the vacuuming is complete, turn off the hand vacuum, keeping the sampler case vertical.

3.5 Sample Recovery

- Raise the humidity in the sampler case (body) by slowly blowing three breaths into the nozzle using the separator as shown in Figure F-6. (Each sampling team member performing the sampling job should have his or her own personal separator.) Tap the sampler case three times with a small rod such as a screwdriver.

- Carefully unscrew the sampling container, while keeping the sampler case vertical, and carefully remove the container.

- Screw the sample catch container cap onto the container.

- Reweigh the container and record data on test data form. Again, weigh the container twice or as many times as necessary until weighings agree within ± 2 mg.

- Place the sampling container in a ziplock plastic bag, along with extra duplicate barcode labels for use by analyst.

- Remove the vinyl gloves and dispose of them in the black trash bag.

Project 9802
WA #55

**Data Entry Sheets
for
Sampler Tests**

Test Sequence Number ____________
Date ____________
Operator ____________

Test Identification

Sampler	______	(Blue Nozzle, CAPS, HVS3 or WIPE)
Substrate	______	(TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in	______	(Yes, No)
Dust Amount	______	(100, 400 mg/ft^2)
Pb Conc	______	(Low, High)
Dust Size	______	(>53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team	______	(Number 1 or 2)
Square number	______	(1, 2, 3 or 4) 1=first, 3=last for carpet and upholstery, else 4=last)

Procedure

Perform the tests according to the sampler test sequence in Appendix Q, and procedures in Appendix E, F, G or H. Housevac A will be used to vacuum the first square before sampler tests, or to vacuum the last square after sampler tests.

If first square:
- Tare weigh bag (run free for 40 seconds, cool 2 minutes, brush and record weight after 1 more minute)
- Vac square for 40 seconds with Housevac A
- Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)

Deposit dust in specified square and weigh the amount deposited (Grind-in dust if applicable)
Sample dust according to the appropriate protocol, weigh the dust collected (except for wipes)
Prepare the dust sample for analysis
If last square:
- *Tare weigh bag (run free for 120 seconds, cool 2 minutes, brush and record weight after 1 more minute)*
- Vac square for 120 seconds with Housevac A
- Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)

Vacuum dust from wand and brush (no weighing)

		Weight of Dust (Balance #________)			Weight of Bag (Balance #________)	
	Total Wt. gm.	Final Wt. gm.	Net Wt. gm.	Time	Weight gm.	Increase gm.
Initial weight of bag (if first or last square)	.0				______	
Vacuum and reweigh bag (if first square)	.1				______	______
Dust deposited	.2______	______	______	______		
Dust collected by sampler (exclu wipes)	.3______	______	______			
Vacuum & reweigh bag (if last square)	4.				______	______

Bar Code for Sample	Bar Code for Blank

NOTE: Submit one blank for each sampler, once each week

Sample relinquished by ____________
Sample received by ____________
Date of transfer ____________

Reviewed by ____________
Date reviewed ____________

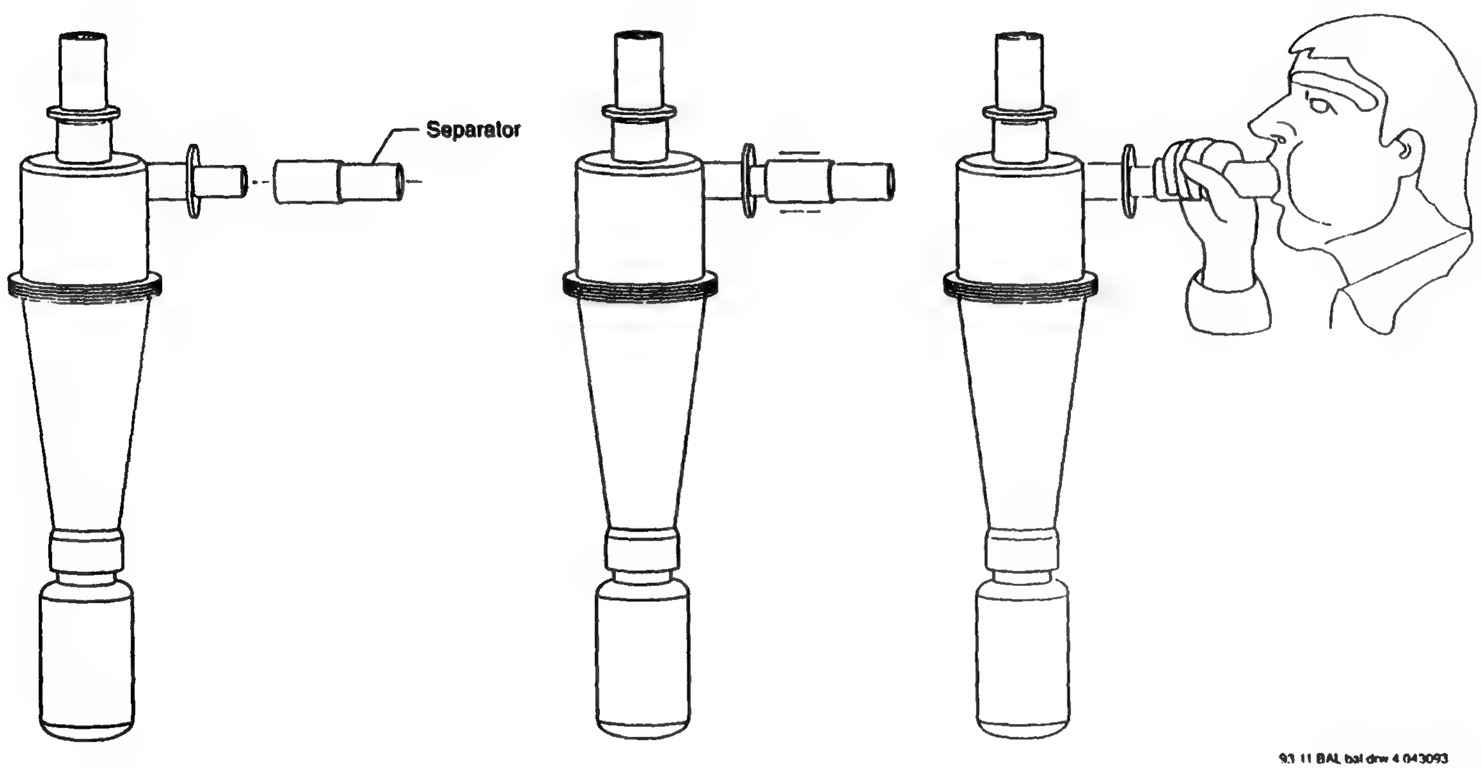

Figure F-6. Illustration of method used to raise humidity in the sampler case.

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4.0 CONTAMINATION AVOIDANCE

The following work practices will be instituted to prevent cross-contamination between samples.

- Clean vinyl gloves (powderless) will be donned prior to collecting each vacuum sample and will be disposed of after each sample is collected.

- The vacuum nozzle will be cleaned with soapy water or Wash-a-bye Baby™ brand disposable wet wipes between each sampling. Vinyl gloves will be used when cleaning nozzles and changed to a clean pair prior to collecting samples. There should be an adequate supply of clean nozzles to accommodate all the vacuum samples collected in one day.

- The templates will be cleaned with a Wash-a-bye Baby™ brand disposable wet wipe between each sample.

5.0 DEVIATIONS FROM SAMPLING PROTOCOLS

Every attempt shall be made to follow this sampling protocol. Deviations from the sampling protocols may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual conditions encountered in the laboratory).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the MRI work assignment leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation should be recollected in accordance with the specified protocol prior to leaving the site.

Deliberate deviations from the sampling protocol must be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA and MRI. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be

expedited. The MRI work assignment leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals from EPA and MRI.

The work assignment leader shall be notified by the sampling team when conditions found in the laboratory do not allow full compliance with the protocol or when the protocol does not appear to apply to the situation. The condition/situation shall be fully documented in a laboratory notebook. The sampling team leader will in turn notify the MRI work assignment leader.

APPENDIX G

PROTOCOL FOR VACUUM SAMPLING OF DUST WITH THE CAPS CYCLONE SAMPLER VAC

PROTOCOL FOR VACUUM SAMPLING OF DUST WITH THE CAPS CYCLONE SAMPLER VAC

1.0 INTRODUCTION

Vacuum samples of dust will be collected from floors (carpeted or uncarpeted) and upholstery, using different research sampling vacuums, one of which is the CAPS cyclone described in this protocol and shown in Figure G-1.

Each 1-ft^2 section to be sampled will be vacuumed in overlapping passes (Figure G-2). A 1-ft^2 template will be used to define the areas to be vacuumed.

2.0 SAMPLING EQUIPMENT AND SUPPLIES

- Cyclone dust collectors
- PVC tubing
- Plastic bottles for collection of dust samples
- PVC nozzles (1 in. in diameter)
- 1-ft^2 templates (full square)
- Steel measuring tape
- Tweezers

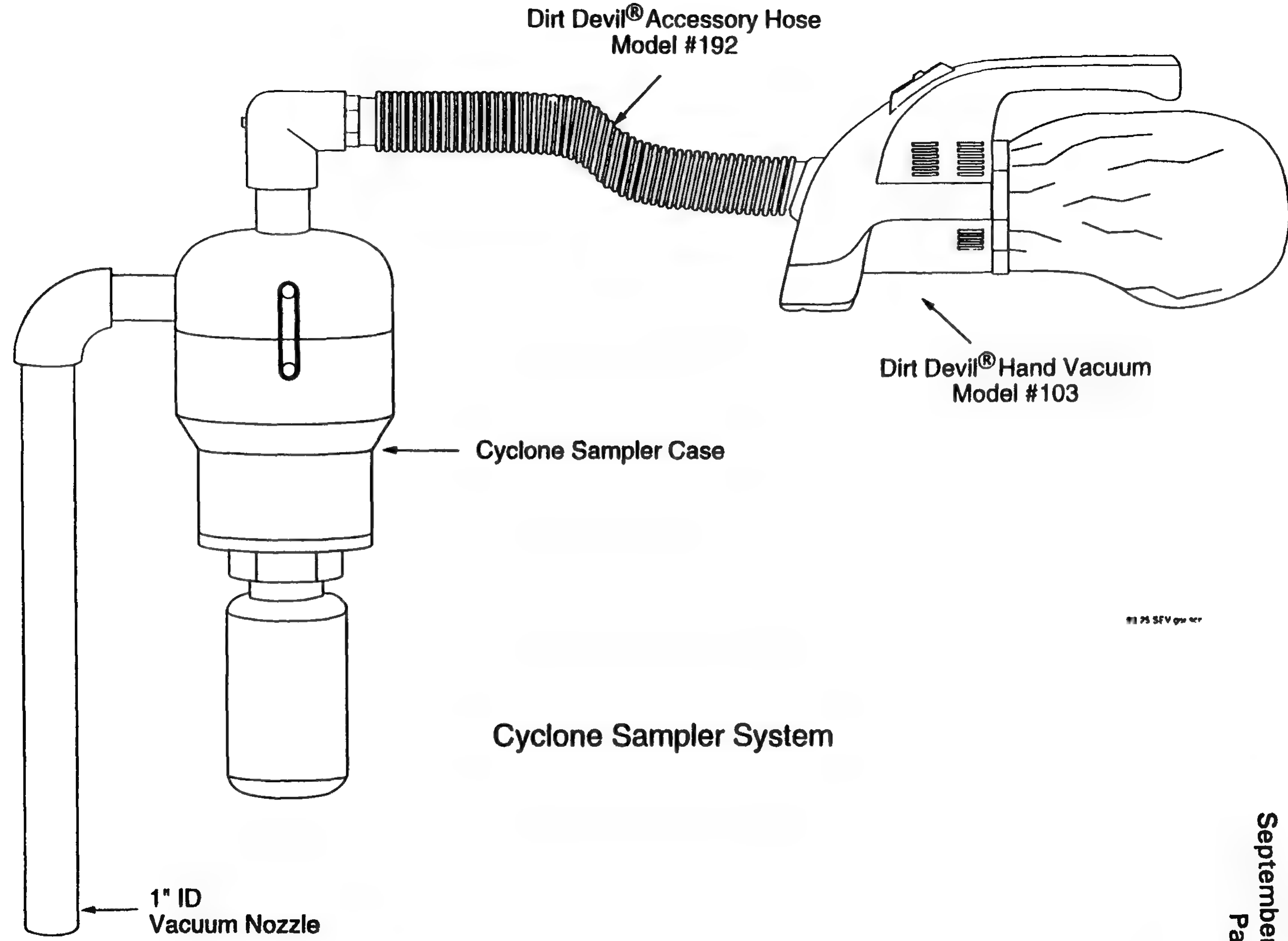

Figure G-1. CAPS cyclone dust collector.

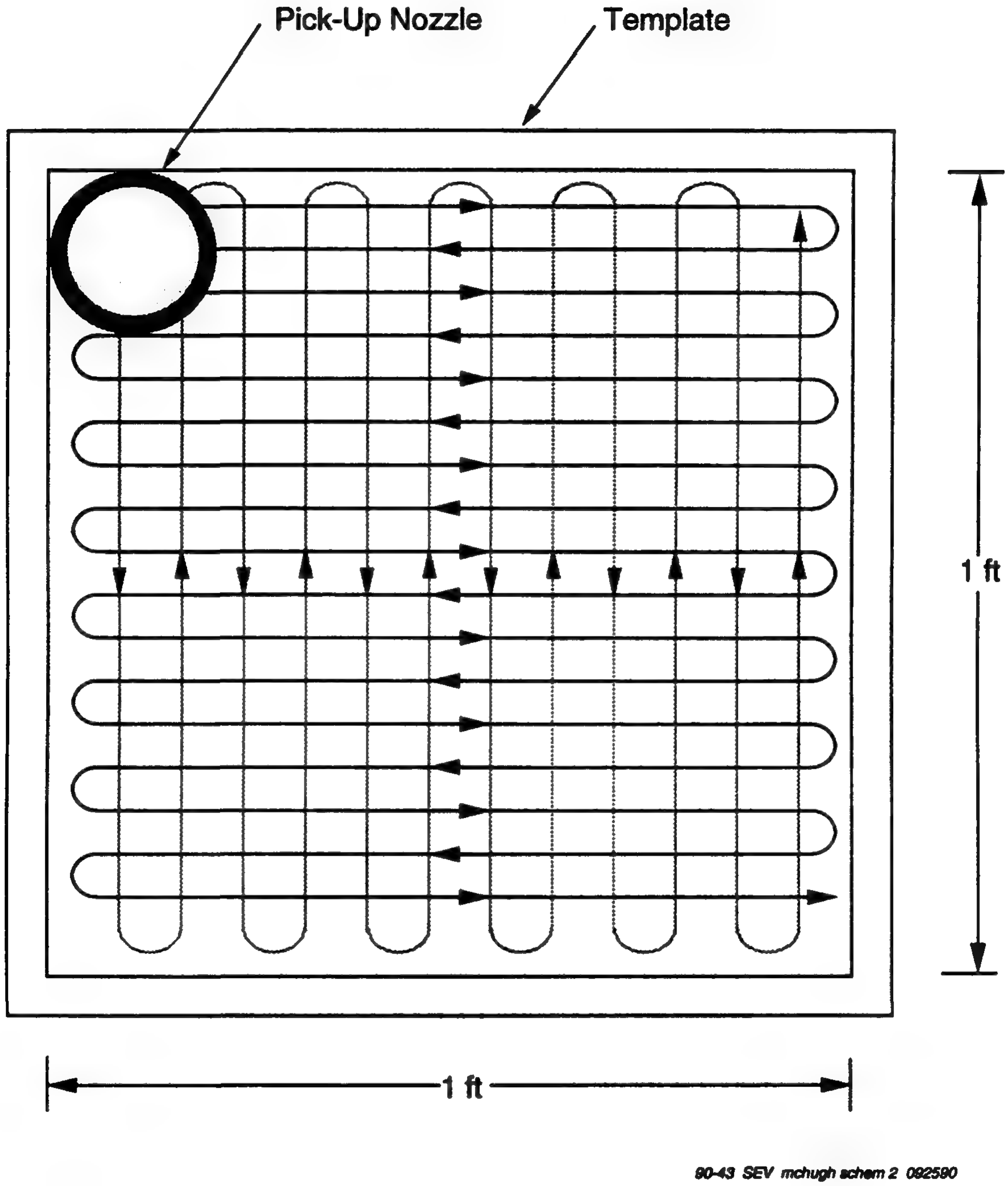

Figure G-2. Vacuum sampling pattern.

- Timing device (stopwatch, timer, or watch with second hand)
- Barcode labels
- 1-qt and 1-gal ziplock plastic bags
- Sample logs (test forms)
- Vinyl gloves (powderless)
- Wash-a-bye Baby premoistened, disposable wipes to clean equipment
- Spatula

3.0 VACUUM SAMPLING PROTOCOL

The following protocol will be used to collect vacuum samples of dust:

3.1 Sampling Preparations

- A clean, 1-ft^2 template will be used to define and measure the surface area to be vacuumed. Measurements will be made after the sample has been collected.
- Record test number, date, time, etc., on the sampling data form.
- Prepare the cyclone dust collector (Figure G-3 as follows):
- Remove the plug (at bottom of cyclone sampler case) by unscrewing it. Set the holder plug aside.
- Remove the three O-rings that hold the dust collector case's top and body together. Set these aside with the holder plug.
- Separate the dust collector's top from its case.

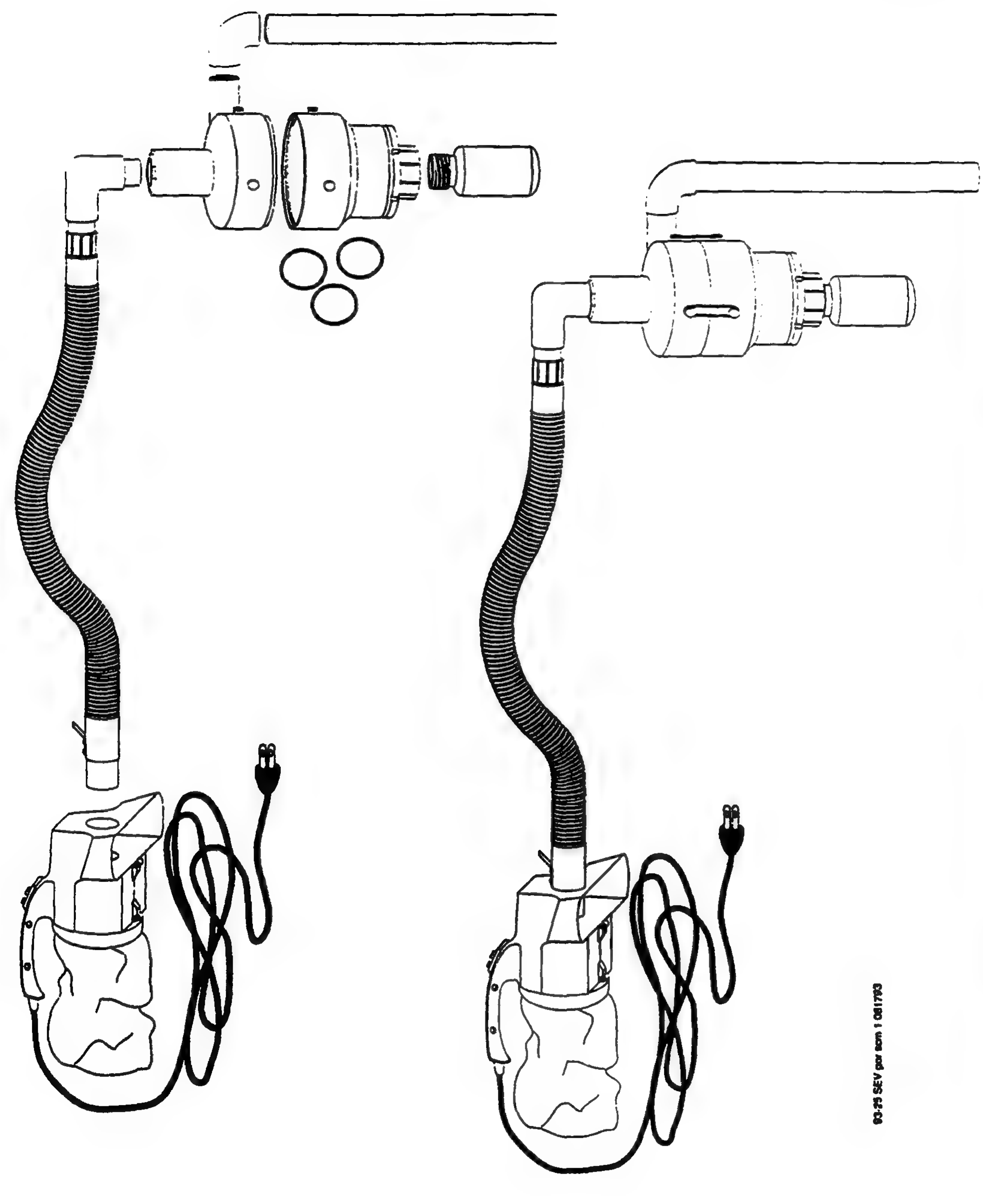

Figure G-3. CAPS cyclone dust collector, assembled and disassembled.

- Wipe the inside surfaces of the dust collector's top and case with Wash-a-bye Baby wipes. Use more than one, if necessary.

- Place the used wipes in a waste container.

- Reassemble cyclone top and sampler case by placing the top onto the sampler case and affixing the three O-rings. Be sure the O-ring holders on the top are aligned with those on the sampler case.

- Affix the hand vacuum to the cyclone sampler case as shown in Figure G-3.

- Don a pair of powderless vinyl gloves prior to handling sample bottles.

- Obtain a sampling dust container (plastic bottle with lid). Affix a barcode label to the container and an identical label on the associated sampling data form.

- Weigh the sample container (with label and lid) and record weighings on test form. Weigh the container twice or as many times as necessary until two weighings agree within ± 2 mg.

- Screw sample container into the bottom of the cyclone. (Store lid in plastic bag.)

- Retrieve a clean 90-degree elbow and a clean 1-in i.d. nozzle from their containers. Attach the elbow (Figure G-4) to the sampler case's 1-in inlet.

- Place the 1-in i.d. nozzle into the open end of the 90-degree elbow (Figure G-4).

- Position the nozzle and the sample case vertically as shown in Figure G-4.

3.2 Conduct Sampling

- Plug the hand vacuum in a 110-V AC outlet.

- Turn on the pump and vacuum the 1-ft^2 area evenly in overlapping passes (at least 50% overlap), first left to right, then front to back over the entire designated area (Figure G-2). Vacuum the area again using this same pattern. For the 1-ft^2 area, vacuuming should not exceed 2 min.

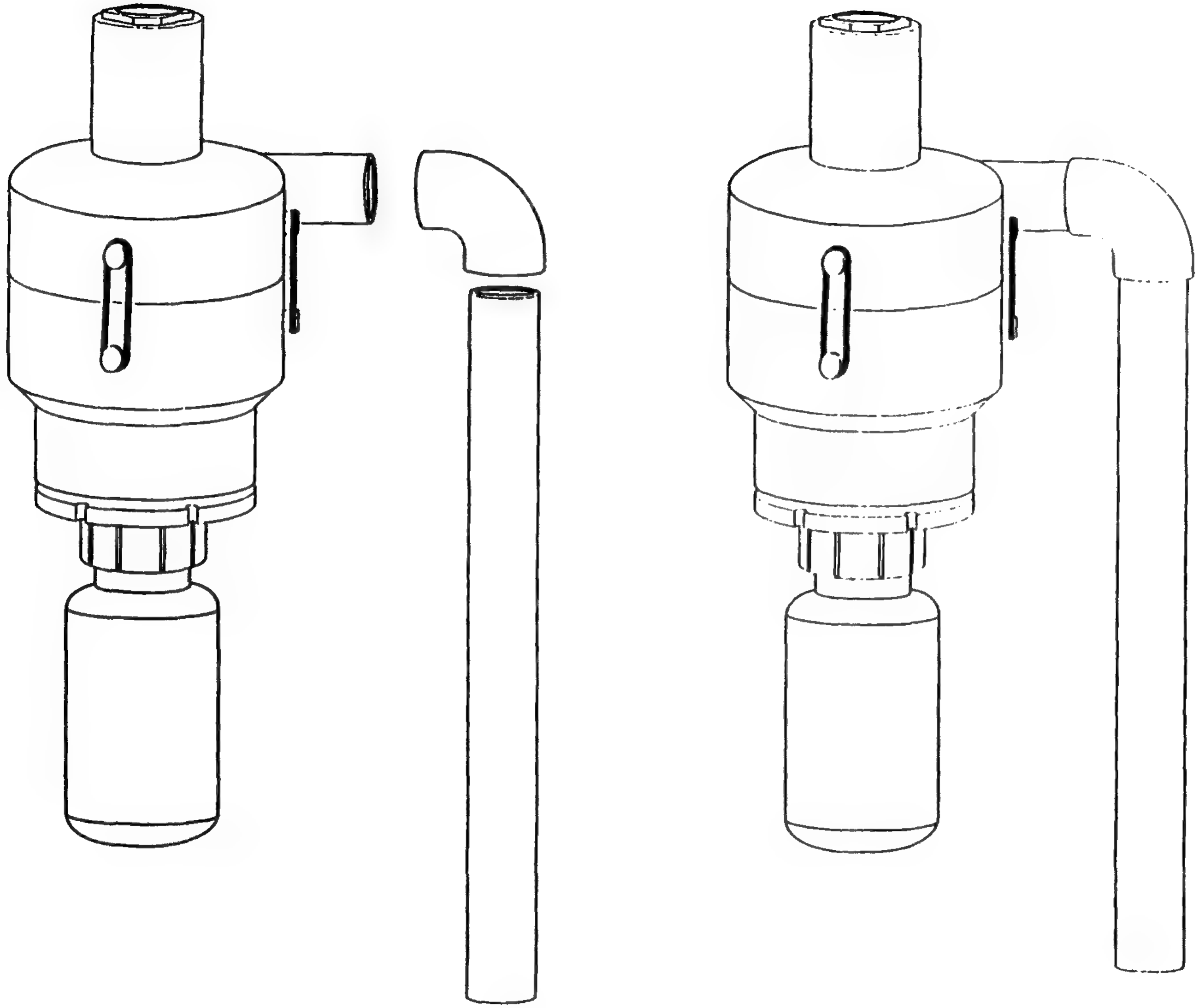

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Figure G-4. Affixing nozzle to cyclone dust collector.

NOTE: The cyclone sampler case must be held vertically throughout the vacuuming process through the removal of the sample container.

NOTE: The template that is used to define a surface area to be vacuumed is the potential source of cross-contamination between samples. The template must be thoroughly cleaned with disposable wipes between each sample.

- When the vacuuming is complete, turn off the hand vacuum, keeping the sampler case vertical.

3.3 Sample Recovery

- Raise the humidity in the sampler case (body) by slowly blowing three breaths into the nozzle using the separator as shown in Figure G-5. (Each sampling team member performing the sampling job should have his own personal separator.) Tap the sampler case three times with a small rod (a screwdriver is an example).

- Carefully unscrew the sample bottle while keeping the sampler case vertical. Replace the lid.

- Reweigh the sample bottle and record on test data form. Again, weigh the bottle twice or as many times as necessary until weighings agree within ± 2 mg.

- Place the sample bottle in a ziplock plastic bag with extra duplicate barcode labels.

- Remove the vinyl gloves and dispose in the black trash bag.

4.0 CONTAMINATION AVOIDANCE

The following work practices will be instituted to prevent cross contamination between samples.

- Clean vinyl gloves (powderless) will be donned prior to collecting each vacuum sample and will be disposed of after each sample is collected.

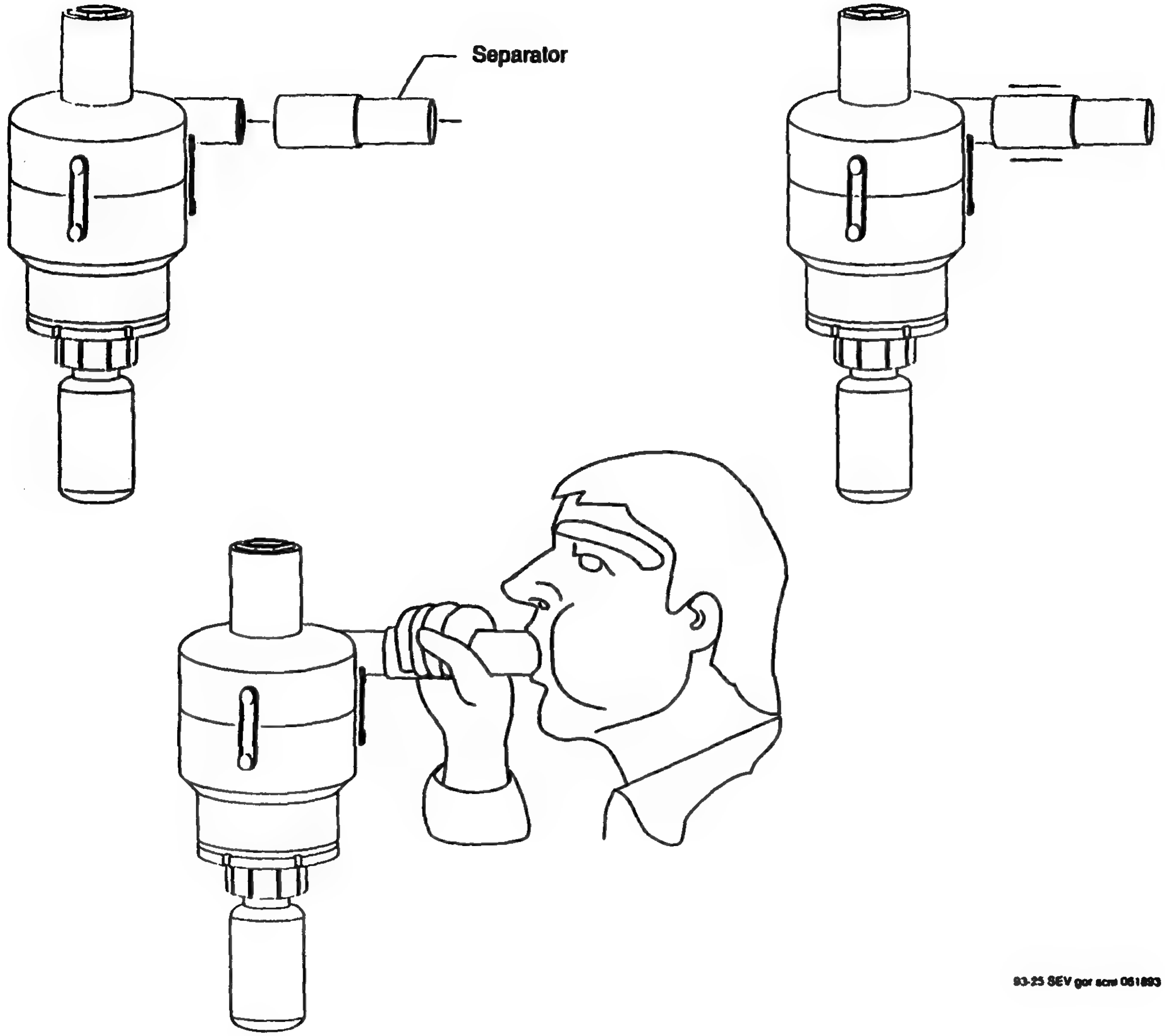

Figure G-5. Separator and its use.

- Sample containers will not be handled without the use of vinyl gloves to prevent the deposition of residues that may interfere with gravimetric analysis of the sample.

- The vacuum nozzle is cleaned with soapy water or "Wash-a-bye Baby" brand disposable wet wipes between each sample. Vinyl gloves will be used when cleaning nozzles and changed to a clean pair prior to collecting samples. There should be an adequate supply of clean nozzles to accommodate all the vacuum samples collected.

- The templates will be cleaned with a "Wash-a-bye Baby" brand disposable wet wipe between each sample.

5.0 DEVIATIONS FROM SAMPLING PROTOCOLS

Every attempt shall be made to follow this sampling protocol. Deviations from the sampling protocols may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories; inadvertent deviations (procedural errors), and deliberate deviations (modifications to the protocol in response to unusual conditions encountered in the laboratory).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the MRI work assignment leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation should be recollected in accordance with the specified protocol prior to leaving the site.

Deliberate deviations from the sampling protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA and MRI. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can by expedited. The MRI work assignment leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals from EPA and MRI.

The work assignment leader shall be notified by the sampling team when conditions found in the laboratory do not allow full compliance with the protocol or when the protocol does not appear to apply to the situation. The condition/situation shall be fully documented in a laboratory notebook. The team leader will in turn notify the MRI work assignment leader.

**Data Entry Sheets
for
Sampler Tests**

Test Sequence Number ____________
Date ____________
Operator ____________

Test Identification

Sampler	______	(Blue Nozzle, CAPS, HVS3 or WIPE)
Substrate	______	(TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in	______	(Yes, No)
Dust Amount	______	(100, 400 mg/ft^2)
Pb Conc	______	(Low, High)
Dust Size	______	(>53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team	______	(Number 1 or 2)
Square number	______	(1, 2, 3 or 4) 1=first, 3=last for carpet and upholstery, else 4=last)

Procedure
Perform the tests according to the sampler test sequence in Appendix Q, and procedures in Appendix E, F, G or H. Housevac A will be used to vacuum the first square before sampler tests, or to vacuum the last square after sampler tests.
If first square:
  Tare weigh bag (run free for 40 seconds, cool 2 minutes, brush and record weight after 1 more minute)
  Vac square for 40 seconds with Housevac A
  Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)
Deposit dust in specified square and weigh the amount deposited (Grind-in dust if applicable)
Sample dust according to the appropriate protocol, weigh the dust collected (except for wipes)
Prepare the dust sample for analysis
If last square:
  Tare weigh bag (run free for 120 seconds, cool 2 minutes, brush and record weight after 1 more minute)
  Vac square for 120 seconds with Housevac A
  Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)
Vacuum dust from wand and brush (no weighing)

		Weight of Dust (Balance #________)				Weight of Bag (Balance #________)	
		Total Wt. gm.	Final Wt. gm.	Net Wt. gm.	Time	Weight gm.	Increase gm.
Initial weight of bag (if first or last square)	.0					______	
Vacuum and reweigh bag (if first square)	.1					______	______
Dust deposited	.2	______	______	______	______		
Dust collected by sampler (exclu wipes)	.3	______	______	______			
Vacuum & reweigh bag (if last square)	4.					______	______

Bar Code for Sample	Bar Code for Blank

NOTE: Submit one blank for each sampler, once each week
Sample relinquished by ____________ Reviewed by ____________
Sample received by ____________ Date reviewed ____________
Date of transfer ____________

APPENDIX H

PROTOCOL FOR VACUUM SAMPLING OF DUST WITH THE BLUE NOZZLE SAMPLER VAC

PROTOCOL FOR VACUUM SAMPLING OF DUST WITH THE BLUE NOZZLE SAMPLER VAC

1.0 INTRODUCTION

Vacuum samples of dust will be collected from floors (carpeted and uncarpeted) and upholstery material using different sampling vacs, one of which is the Blue Nozzle Sampler described in this protocol and shown in Figure H-1. This vacuum sampling device consists of a Teflon pickup nozzle mounted on a preweighed 37-mm, mixed cellulose ester filter cassette (0.8-μm pore size). This collection device is coupled to a Gast rotary-vane vacuum pump with Tygon tubing (Figure H-1).

Each 1-ft^2 section of the surface to be sampled will be vacuumed in overlapping passes (Figure H-2). A 1-ft^2 template will be used to define the area to be vacuumed.

2.0 SAMPLING EQUIPMENT AND SUPPLIES

- Gast rotary-vane vacuum pumps
- Tygon tubing
- Gelman GN-4, 37-mm, mixed cellulose ester (MCE) filter cassettes (0.8-μm pore size)
- Teflon pickup nozzles
- 1-ft^2 templates
- Steel measuring tape
- Screwdriver
- Barcode labels
- 1-qt and 1-gal ziplock plastic bags
- Plastic trash bag
- Sample data sheets
- Vinyl gloves (powderless)

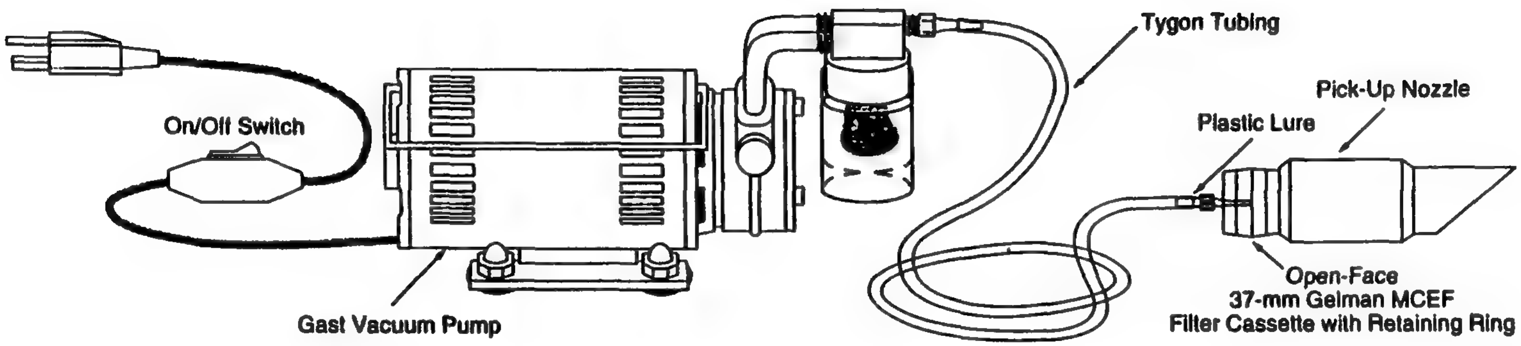

Figure H-1. Schematic of rotary vacuum pump with blue nozzle.

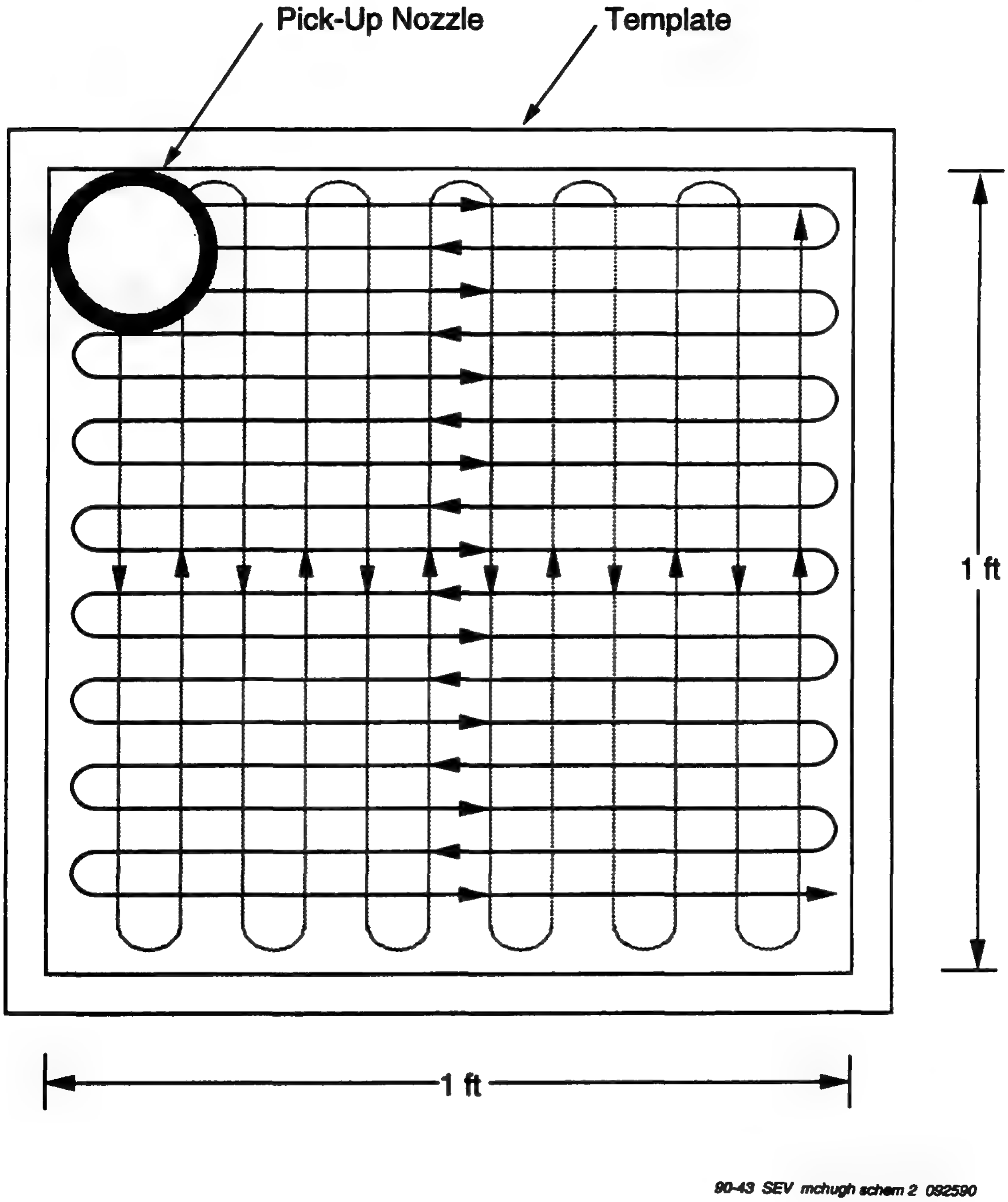

Figure H-2. Vacuum sampling pattern.

3.0 VACUUM SAMPLING PROTOCOL

The following protocol will be used to collect vacuum samples of dust using the blue nozzle sampler.

3.1 Sampling Preparations

- A clean, 1-ft^2 template will be used to define the surface area to be vacuumed.

  NOTE: The template that is used to define a surface area to be vacuumed is a potential source of cross contamination between samples. The template must be thoroughly cleaned with disposable wipes between each sample.

- Record test number, date, time, etc., on the sampling data form.

- Don a pair of powderless vinyl gloves prior to handling filter cassettes. <u>Do not touch the cassettes with bare hands.</u>

- Obtain one of the filter cassettes from the box in the laboratory so that the cassette will have equilibrated with conditions in the test laboratory.

- Weigh the cassette and record weighings on data form. Weigh the cassettes twice or as many times as necessary until two weighings agree within ± 2 mg.

- Pry open the top section of the filter cassette with a clean flat-edged screwdriver or equivalent tool. Carefully remove the top section. (See Figure H-3)

  NOTE: The middle retaining ring holds the filter and support pad in place against the bottom section of the cassette (Figure H-3). The retaining ring should be inspected to ensure that it is seated tightly against the bottom section. If the middle ring is not secure, the filter may tear during the sampling procedure. The seal between the bottom section of the cassette and the middle ring can be secured by squeezing the two sections firmly between the index fingers and the thumbs of both hands.

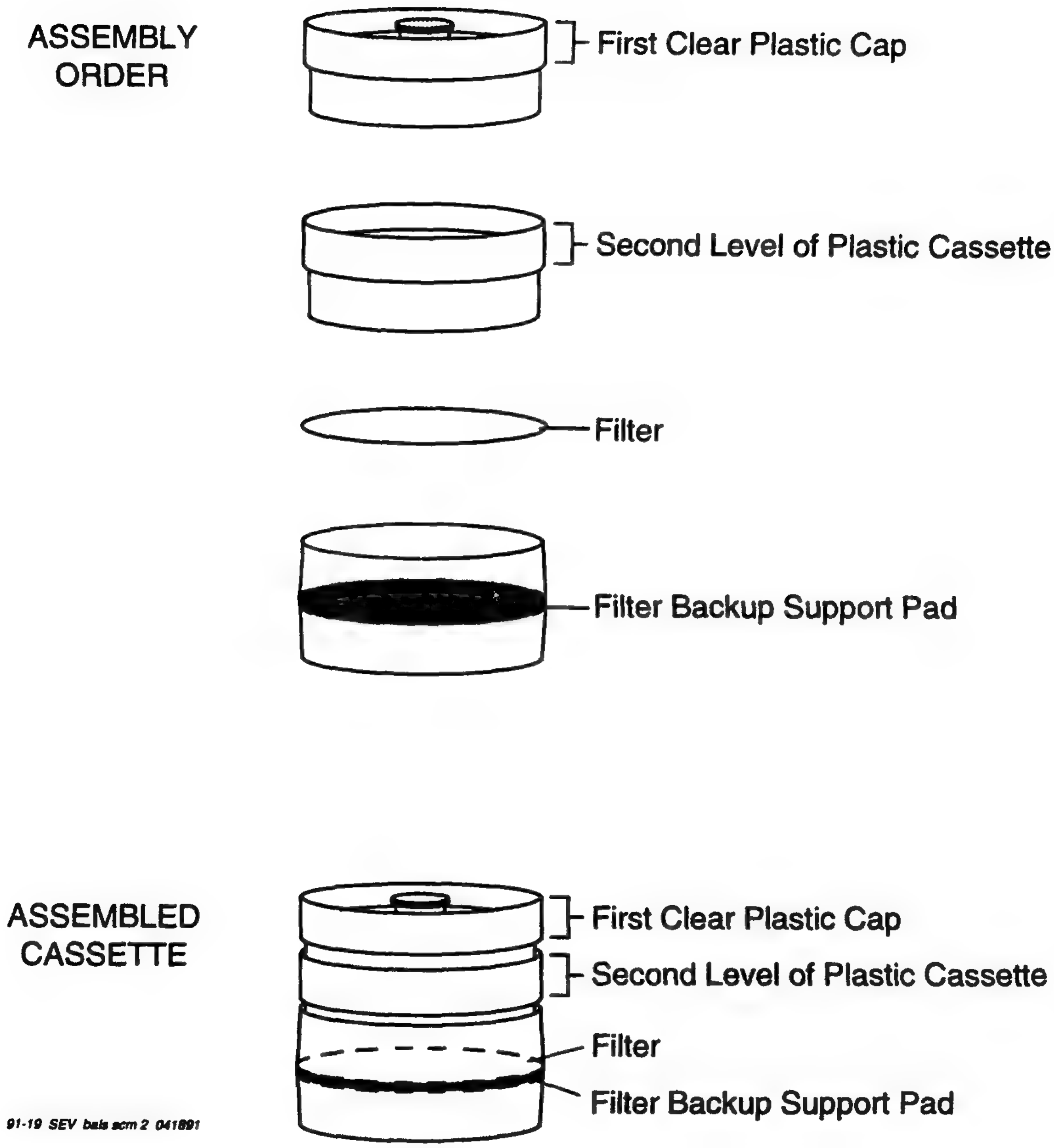

Figure H-3. Unassembled and assembled dust sample cassette.

- Store the top section of the cassette inside a ziplock bag during sampling to avoid contamination.

- Attach Tygon tubing between the inlet of the pump and the outlet of the filter cassette.

- Insert the open side of the filter cassette into a pickup nozzle. The nozzle must fit snugly around the rim of the retaining ring. If it does not fit snugly, replace the nozzle or discard the filter cassette.

3.2 Conduct Vacuuming

- Turn on the pump and vacuum the 1-ft^2 area in overlapping passes (at least 50 percent overlap), first left to right and then front to back over the entire designated area (see Figure H-2). Vacuum the designated area again using this same pattern. For the 1-ft^2 area, vacuuming should not exceed 2 min.

- When the vacuuming is complete, hold the open end of the nozzle upright, and turn off the pump.

3.3 Sample Recovery

- While maintaining the filter cassette in an upright position, carefully remove the nozzle and disconnect the Tygon tubing.

- Replace the top section of the cassette.

- Reweigh the cassette and record weight on test data form. Again, weigh the cassette twice or as many times as necessary until weighings agree within ± 2 mg.

- Place the filter cassette in a ziplock plastic bag. Attach barcode label to plastic bag and duplicate barcode label on the sampling data form.

- Place the plastic bag containing the cassette inside a second plastic bag along with extra duplicate barcode labels for use by analyst.

- Remove the vinyl gloves and dispose in the trash bag.
- Store samples in a clean container until ready for analysis.

4.0 PREPARATION OF SAMPLING BLANK SAMPLES

Sampling blank samples will consist of a filter cassette that is handled in the same manner as the regular vacuum samples, except that no sample is collected. Provide a blank sample with each batch of samples, or once a week at minimum.

5.0 CONTAMINATION AVOIDANCE

The following work practices will be utilized to prevent contamination of samples.

- Clean vinyl gloves (powderless) will be donned prior to collecting each vacuum sample and will be disposed of after each sample is collected.
- Filter cassettes must not be handled without the use of vinyl gloves to prevent the disposition of residues that may interfere with gravimetric analysis of the sample. If the filter cassette is inadvertently touched prior to collecting the sample, the filter cassette will be discarded.
- The vacuum nozzle will be cleaned with soapy water or "Wash-a-bye Baby" brand disposable wet wipes between each sample. There should be an adequate supply of clean nozzles to accommodate all the vacuum samples collected.
- The templates will be cleaned with a "Wash-a-bye Baby" brand disposable wet wipe between each sample.

6.0 DEVIATIONS FROM SAMPLING PROTOCOLS

Every attempt shall be made to follow this sampling protocol. Deviations from the sampling protocols may compromise the data quality and completeness objectives of the project. Deviations from the protocols will generally fall into two categories: inadvertent deviations (procedural errors), and deliberate deviations (modifications to the protocol in response to unusual conditions encountered in the laboratory).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sample log and immediately notify the MRI work assignment leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, samples affected by the inadvertent deviation should be recollected in accordance with the specified protocol.

Deliberate deviations from the sampling protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA and MRI. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be expedited. The MRI work assignment leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals from EPA and MRI.

The work assignment leader shall be notified by the sampling team when conditions found in the laboratory do not allow full compliance with the protocol or when the protocol does not appear to apply to the situation. The condition/situation shall be fully documented in a laboratory notebook.

Data Entry Sheets
for
Sampler Tests

Test Sequence Number ____________
Date ____________
Operator ____________

Test Identification

Sampler	_____	(Blue Nozzle, CAPS, HVS3 or WIPE)
Substrate	_____	(TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in	_____	(Yes, No)
Dust Amount	_____	(100, 400 mg/ft^2)
Pb Conc	_____	(Low, High)
Dust Size	_____	(>53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team	_____	(Number 1 or 2)
Square number	_____	(1, 2, 3 or 4) 1=first, 3=last for carpet and upholstery, else 4=last)

Procedure
Perform the tests according to the sampler test sequence in Appendix Q, and procedures in Appendix E, F, G or H. Housevac A will be used to vacuum the first square before sampler tests, or to vacuum the last square after sampler tests.
If first square:
  Tare weigh bag (run free for 40 seconds, cool 2 minutes, brush and record weight after 1 more minute)
  Vac square for 40 seconds with Housevac A
  Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)
Deposit dust in specified square and weigh the amount deposited (Grind-in dust if applicable)
Sample dust according to the appropriate protocol, weigh the dust collected (except for wipes)
Prepare the dust sample for analysis
If last square:
  Tare weigh bag (run free for 120 seconds, cool 2 minutes, brush and record weight after 1 more minute)
  Vac square for 120 seconds with Housevac A
  Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)
Vacuum dust from wand and brush (no weighing)

		Weight of Dust (Balance #________)				Weight of Bag (Balance #________)	
		Total Wt. gm.	Final Wt. gm.	Net Wt. gm.	Time	Weight gm.	Increase gm.
Initial weight of bag (if first or last square)	.0					______	
Vacuum and reweigh bag (if first square)	.1					______	______
Dust deposited	.2	______	______	______	______		
Dust collected by sampler (exclu wipes)	.3	______	______	______			
Vacuum & reweigh bag (if last square)	4.					______	______

Bar Code for Sample	Bar Code for Blank

NOTE: Submit one blank for each sampler, once each week
Sample relinquished by ____________ Reviewed by ____________
Sample received by ____________ Date reviewed ____________
Date of transfer ____________

APPENDIX I

PROTOCOL FOR VACUUMING AND SAMPLING OF DUST WITH HOUSEVACS

PROTOCOL FOR VACUUMING AND SAMPLING OF DUST WITH HOUSEVACS

1.0 INTRODUCTION

Many of the tests to be done for this study require sampling with Housevacs (four different brands) as specified in Appendix P. This protocol describes the procedure to be used in carrying out the vacuuming operations and sampling of collected dust after the final vacuuming. It is applicable to all substrates: carpeted and uncarpeted floors and upholstery material. For all substrates, the area to be vacuumed will be 6.78 ft^2 (18 x 54 in).

2.0 EQUIPMENT AND SUPPLIES

- Tape measure or ruler
- Template (18 x 54 in)
- Wide masking tape
- Stopwatch
- Dust sample bottles
- Barcode labels
- Test data forms
- Substrates

3.0 PROCEDURE

3.1 Preparations

- Use data entry forms (attached at the end of this appendix).

- Use preconditioned substrates per Appendix D.

- Place template on substrate and mark test area with masking tape.

- Select Housevac to be tested (per test design) and proper cleaning attachment depending on substrate to be tested. **(Note: for carpet, always use the power nozzle attachment.)**

- Measure the outside width of the nozzle housing, in inches.

- Calculate the number of vacuuming "strips" (N) to be used, as follows:

  $$N = \text{Number of strips} = \frac{18 \text{ in}}{\text{nozzle width}} \quad \text{(Round to nearest whole number.)}$$

  Example: If nozzle width is 7 in, the number of "strips" would be 3. Thus, each strip would be 6 in wide.

- Use duct tape to mark the test strips at both ends of the test area (see Figure I-1).

- Determine the stroke pattern to be used, per Figure I-1. In all cases, the total number of strokes used must be 16 (a movement in one direction is one stroke).

3.2 Vacuuming Procedure

- Cleaner nozzle is to be placed on the test carpet so that the front of the nozzle is coincident with the line defining the beginning of the test area and with the right side of the nozzle at the right side boundary shown in the applicable illustration in Figure I-1.

- Carry out vacuuming per the stroke pattern determined above. Each stroke pattern utilizes a total of 16 strokes. Length of time for each stroke should be near 2.5 sec, for a total vacuuming time of 40 sec.

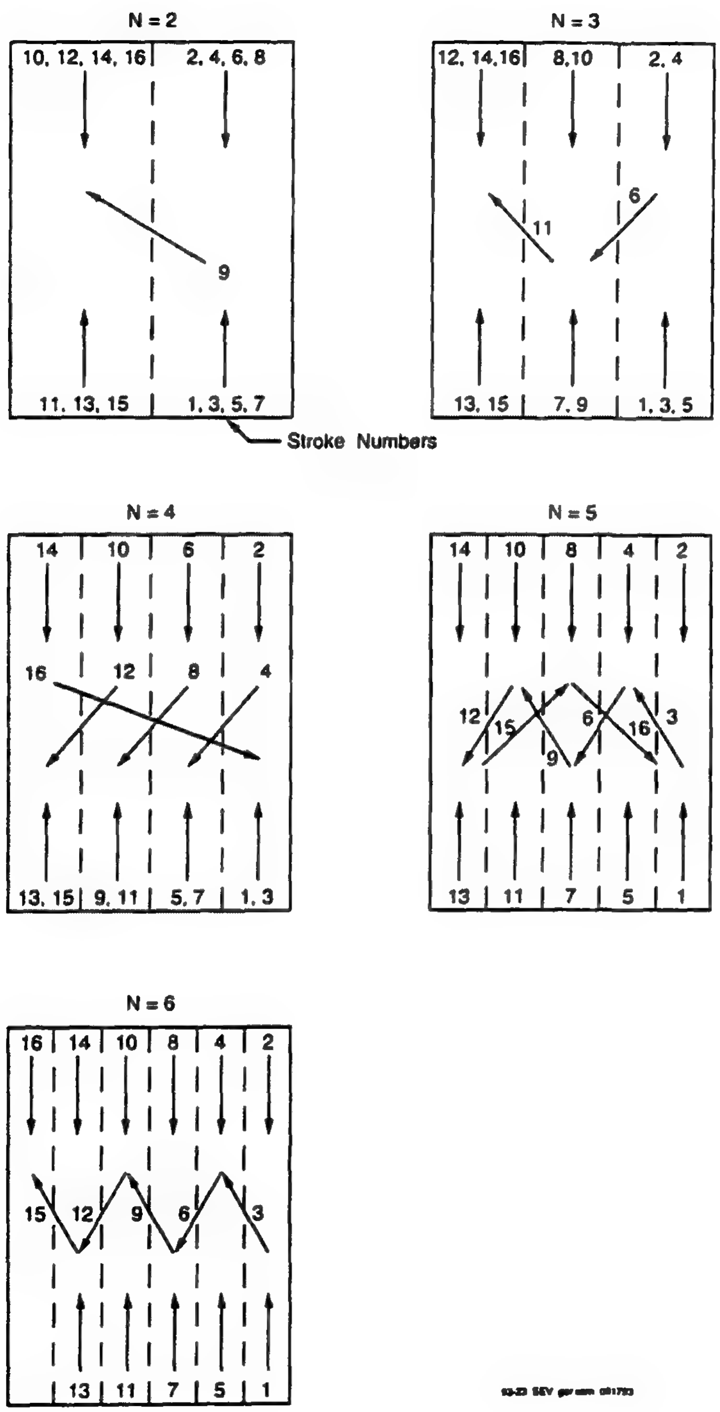

Figure I-1. Test cleaning patterns.

- Ensure that each forward stroke ends with the cleaner nozzle coincident with the end of the test area. When the cleaner reaches the extreme left strip, align the left side of the nozzle with the left side boundary of the test area. Take care to ensure that during each stroke the side of the nozzle, right side or left side as applicable, is kept aligned with the side boundary of the test strip being cleaned, except for cross-over strokes.

- At the end of the last stroke, smoothly tilt or lift the nozzle off the substrate and allow Housevac to run 10 sec to clear the system.

- During this 10-sec period, the hose used on some Housevacs should be flexed to help clear it of any dust.

3.3 Conduct Test

The procedure to be used in conducting each Housevac test is as follows:

- Tare weigh new bag:
  Run free for 40 sec, cool 2 min, brush and record weight after 1 more min.

- Vacuum for 40 sec, before any dust is deposited.

- Reweigh bag (cool 2 min, brush and record weight after 1 more min)

- Deposit dust, vacuum for 40 sec, reweigh bag). Total of 3 times.

- Vacuum and reweigh bag 3 times (no dust deposit)

- Recover dust sample from bag per Section 4.0.

- Vacuum dust from wand and brush (no weighing).

4.0 SAMPLE COLLECTION

After the specified number of vacuumings and weighings, a sample of the dust collected in the Housevac bag will need to be taken for analysis, as follows:

- Obtain one of the wide mouth plastic sample bottles. Place a barcode label on the bottle and an identical label on the test form.

- Weigh the empty bottle (with lid and label). Record weight on test form. Weigh the bottle twice or as many times as necessary until two weighings agree within ±2 mg.

- Carefully cut away any sealing flaps on the bag inlet hole.

- Place the bag over the top of the weighed plastic bottle, with the bag opening facing down into the bottle. If the bag opening is larger than the top of the bottle, use a suitable clean funnel for this operation.

- Tap the outside of the bag; this should cause some dust inside to fall into the sample bottle.

- Place the lid back on the sample bottle and reweigh. Record weight on test form. Again, weigh the bottle twice or as many times as necessary until two weighings agree within ±2 mg.

5.0 CONTAMINATION AVOIDANCE

Contamination refers to both the inadvertent increase (or decrease) in the weight of dust collected by the Housevac (other than that applied to the substrate) and potential Pb contamination of that dust or dust sample.

To avoid such contamination, the Housevac must be run only as specified in the protocol, to avoid "sucking in" any other dust in the vicinity. Care must especially be taken in removing the bag from the Housevac for weighing, so that none of the collected dust escapes or is allowed to fall out of the bag.

For sample collection, always use clean equipment. Collect the sample carefully, and in an area that minimizes potential contamination from other sources or test operations.

6.0 DEVIATIONS FROM PROTOCOL

Every attempt shall be made to follow this protocol. Deviations from the protocol may compromise the data quality and completeness objectives of the project. Deviations from the protocol will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual (or unanticipated conditions).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the MRI work assignment leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, any tests or samples affected by the inadvertent deviation shall be redone in accordance with the specified protocol.

Deliberate deviations from the protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be expedited. The MRI work assignment leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals.

Project 9802
WA No. 55

Data Entry Sheets
for
Housevac Tests

Test Sequence Number ____________
Date ____________
Operator ____________

Test Identification

Housevac ________ (A, B, C or D)
Substrate ________ (TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in ________ (Yes, No)
Dust Amount ________ (100, 400 mg/ft^2
Pb Conc ________ (Low, High)
Dust Size ________ (<53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team ________ (number 1 or 2)

Procedure

Perform the tests according to the housevac test sequence in Appendix P, and vac procedure in Appendix I
Tare weigh new bag:
Run free 40 sec, cool 2 min, brush and record weight after 1 more min
Vacuum for 40 sec before any dust deposit
Reweigh bag (cool 2 min, brush and record weight after 1 more min)
Deposit dust, vacuum 40 sec, weigh the bag. Total of 3 times. (Grind-in after each dust deposit, if applicable)
Repeat vacuuming only (vacuum 40 sec, weigh the bag) 3 times
Shake dust from the bag, weigh, prepare for lead analysis
Vacuum dust from wand and brush (no weighing)

		Weight of Dust (Balance #________)				Weight of Bag (Balance #________)	
		Total Wt gm.	Tare Wt gm.	Net Wt gm.	Time	Weight gm.	Increase gm.
Tare weight of bag	.0					______	
Vacuum and weigh	.1				______	______	______
Add dust, vac & weigh	.2	______	______	______		______	______
Add dust, vac & weigh	.3	______	______	______		______	______
Add dust, vac & weigh	.4	______	______	______		______	______
Vacuum & weigh	.5					______	______
Vacuum & weigh	.6					______	______
Vacuum & weigh	.7					______	______
Dust sent to lab	.8	______	______	______			

Bar Code for Sample	Bar Code for Blank

Submit one blank for each week

Sample relinquished by ________________
Sample received by ________________
Date of transfer ________________

Reviewed by ____________
Date reviewed ____________

APPENDIX J

DIGESTION PROCEDURE

MODIFIED METHOD 3050 FOR ANALYSIS OF LEAD (Pb) IN WIPE DUST SAMPLES

DIGESTION PROCEDURE
MODIFIED METHOD 3050 FOR ANALYSIS OF LEAD (Pb) IN WIPE DUST SAMPLES

1.0 SUMMARY OF METHOD

This analytical method is a modified version of Method 3050 from the SW-846, 3rd Edition manual. It is written in a manner that provides ease of execution relative to the published procedure. This method is used for the acid digestion of wipe dust samples and associated quality control (QC) samples for ICP-AES analysis of lead (Pb). It should be noted that this procedure for preparing dust wipe samples does not use hydrochloric acid for digestion. The final reflux is nitric acid (HNO_3), and the sample digestate will be approximately 10% (v/v) HNO_3 following digestion.

The entire wipe dust sample is digested in HNO_3 and hydrogen peroxide (H_2O_2). The digestates are diluted to final volume following a final reflux and cookdown using nitric acid.

2.0 APPARATUS AND MATERIALS

- Beakers: Griffin 250-mL
- Watch glasses
- Forceps: polyethylene
- Volumetric flasks with stoppers: 100-mL
- Funnels: Plastic, porcelain, or glass fitted with fast filter paper suitable for lead analysis. (Buchner type funnels are recommended due to potentially large amount of undigested wipe materials commonly encountered using this method.)
- Thermometers: red alcohol and covers range of 0° to 110°C
- Hot plates: capable of maintaining a temperature of 80° to 100°C
- Centrifuge
- Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

- Kimwipes™
- Disposable plastic 10-mL syringes with Luer-lok fittings
- Gelman Acrodisc filters, female Luer-lok, 0.45-μm pore size

3.0 REAGENTS

- ASTM Type I water: Minimum resistance of 16.67 megaohm-cm, or equivalent
- Concentrated (70 to 71%) HNO_3: Baker instra-analyzed, or equivalent
- Hydrogen peroxide (30%), reagent grade

4.0 QUALITY CONTROL

For each group of samples processed, sample preparation quality control samples (SP QCs) should be carried throughout the entire sample preparation and analytical process. The SP QCs to include in each batch of samples are summarized in Table J-1.

TABLE J-1. SUMMARY OF SAMPLE PREPARATION QC SAMPLES—WIPES

Sample Type	Description	Frequency	Data Quality Objective
Method Blank	Empty beaker, equivalent to a reagent blank	1 per 20 samples; minimum of 1 per batch	Measured value less than 10 times the Instrumental Detection Limit
Reference Material (2 different lead levels)	1 g ± 0.1 g of reference material plus a blank wipe	1 of NIST SRM 1646 and 1 NIST SRM 2704 per batch	Accuracy of ±25% from average known value
No-Spiked Sample	Blank wipe	1 per 20 samples; minimum of 1 per batch	Not applicable—used to calculate spiked sample recovery
Spiked Sample and spiked duplicate	1 mL of 100 μg/mL lead stock solution plus a blank wipe	1 per 20 samples; minimum of 1 per batch	Accuracy of ±20% from average known value

5.0 PROCEDURE

5.1 Care should be taken during the execution of each step of the following procedure to ensure the sample losses do not occur due to spillage and the gains do not occur due to contamination (See **Note 1**).

NOTE 1: All reagent sources (lot numbers) used for sample preparation must be recorded in a laboratory notebook. In addition, any inadvertent deviations to this procedure, unusual occurrences or observations must also be recorded on a real-time basis as samples are processed. The laboratory task leader must be informed in a manner which permits as close to real-time action as possible if any deviations or unusual happenings occur in order to take any needed corrective actions.

All samples in a processing batch must be treated equally. For example, if one sample requires additional hydrogen peroxide, then all samples in that batch must be given additional peroxide.

5.2 Document the condition of samples received in a laboratory note.

5.3 Label 250-mL Griffin beakers for each wipe sample and associated quality control sample to be processed.

5.4 The wipe samples do not require weighing by the LAB. Carefully open the container containing the sample and transfer the contents into labeled Griffin beakers using a new pair of plastic gloves and\or plastic forceps (see **Note 2**).

NOTE 2: For spikes and spike duplicates, transfer blank wipe to beakers targeted for these samples and spike onto them according to details in Table J-1.

5.5 SAMPLE DIGESTION

Perform digestion of each sample as follows:

Carefully, add 25 mL of 1:1 HNO_3 to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to 85° to 100°C and reflux for 10 to 15 min without boiling.

5.5.2 Allow the sample to cool.

5.5.3 Add 10 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 min without boiling.

5.5.4 Repeat this last step (5.5.3) to ensure complete oxidation.

5.5.5 Remove watch glass and allow the solution to evaporate to approximately 10 mL without boiling, while maintaining a covering of solution over the bottom of the beaker (see Note 3).

NOTE 3: Because ribbed watch glasses are not available, the watch glasses may be removed to accelerate volume reduction while ensuring minimal contamination of the samples through careful observation. When removing these watch glasses, care must be exercised to avoid losses by rinsing them with a minimum amount of Type I water (rinsed into the sample beaker) and avoiding contaminating them by placing them upside down on new clean Kimwipes®.

5.5.6 Allow the sample to cool.

5.5.7 Add 5 mL Type I water and 5 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

5.5.8 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged (see Note 4).

NOTE 4: Do not add more than a total of 10 mL of 30% H_2O_2 even if effervescence has not been reduced to a minimal level.

5.5.9 Remove the watch glass and continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 10 mL (see Note 3).

5.5.10 Allow the digestates to cool, rinse the beaker walls and bottom of the watch glass into the digestate, and quantitatively transfer to a 100-mL volumetric flask (see **Note 5**). Dilute to volume with Type I water.

NOTE 5: It is recommended that a funnel be used to aid in quantitative transfer due to the potential presence of large quantities of undissolved wipe material. Use either a clean porcelain funnel or a glass or plastic funnel. If the funnel contains small premade holes (Buchner type), then no filter needs to be used. Otherwise fit the funnel with the fastest filter paper suitable for metals analysis filtering.

5.6 Particulates in the digestate then should be removed by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement. A disposable syringe equipped with an Acrodisc filter can be used to filter a portion of the sample digest prior to analysis.

5.7 The diluted digestate solution contains approximately 10% (v/v) HNO_3. Calibration standards used for instrumental measurement should be made with this level of HNO_3.

APPENDIX K

DIGESTION PROCEDURE

MODIFIED METHOD 3050 FOR ANALYSIS OF LEAD (Pb) IN VACUUM DUST SAMPLES (CASSETTES OR BOTTLES) COLLECTED WITH A SAMPLER OR HOUSEVAC

DIGESTION PROCEDURE
MODIFIED METHOD 3050 FOR ANALYSIS OF LEAD (Pb) IN VACUUM DUST SAMPLES (CASSETTES OR BOTTLES) COLLECTED WITH A SAMPLER OR HOUSEVAC

1.0 SUMMARY OF METHOD

This analytical method is a modified version of Method 3050 from the SW-846, 3rd Edition manual. It is written in a manner which provides ease of execution relative to the published procedure. This method is used for the acid digestion of dust samples in cassettes or bottles and associated quality control (QC) samples for ICP-AES analysis of Pb. The primary focus of the modifications relative to the published procedure is a change in amounts of reagents used and a change in final dilution volume to accommodate the sample size. In addition, the method has been modified for handling the samples contained in filter cassettes and bottles. It should be noted that this procedure does not use hydrochloric acid for digestion. The final reflux is nitric acid (HNO_3) and the sample digestate will be approximately 10% (v/v) HNO_3 following digestion.

The samples are digested in HNO_3 *and hydrogen peroxide* (H_2O_2). The digestates are diluted to final volume following a final reflux and cookdown using nitric acid.

2.0 APPARATUS AND MATERIALS

- Beakers: Griffin 100-mL
- Watch glasses
- Forceps: polyethylene
- Volumetric flasks with stoppers: 25-mL
- Thermometers: red alcohol and covers range of 0° to 110°C
- Hot plates: capable of maintaining a temperature of 80° to 100°C
- Centrifuge
- Centrifuge tubes: polyethylene with screw caps, 50-mL capacity
- Kimwipes™
- Disposable plastic 10-L syringes with Luer-lok fittings

- Gelman Acrodisc filters, female Luer lok, 0.45-μm pore size
- Screwdriver

3.0 REAGENTS

- ASTM Type I water: Minimum resistance of 16.67 megaohm-cm, or equivalent
- Concentrated (70 to 71%) HNO_3: Baker instra-analyzed, or equivalent
- Hydrogen peroxide (30%), reagent grade

4.0 QUALITY CONTROL

For each group of samples processed, sample preparation quality control samples (SP QCs) should be carried throughout the entire sample preparation and analytical process. The SP QCs to include in each batch of samples are summarized in Table K-1.

5.0 PROCEDURE

5.1 Care should be taken during the execution of each step of the following procedure to ensure that sample losses do not occur due to spillage and that gains do not occur due to contamination (see **NOTE 1**).

NOTE 1: All reagent sources (lot numbers) used for sample preparation must be recorded in a laboratory notebook. In addition, any inadvertent deviations to this procedure, unusual occurrences or observations must also be recorded on a real-time basis as samples are processed. The laboratory task leader must be informed in a manner which permits as close to real-time action as possible if any deviations or unusual happenings occur in order to take any needed corrective actions.

All samples in a processing batch must be treated equally. For example, if one sample requires additional hydrogen peroxide, then all samples in that batch must be given additional peroxide.

TABLE K-1. SAMPLE PREPARATION QC SAMPLES—CASSETTES AND DUST IN BOTTLES

Sample Type	Description	Frequency	Data Quality Objective
Method Blank	Empty beaker, equivalent to a reagent blank and a no-spiked sample for dust in bottles.	1 per 20 cassette plus dust bottle samples; minimum of 1 per batch	Measured value less than 10 times the Instrumental Detection Limit
Reference Material (2 different lead levels)	1 g ± 0.1 g of reference material into an empty beaker	1 NIST SRM 1646 and 1 NIST SRM 2704 per batch	Accuracy of ±25% from average known value
No-Spiked Sample, cassette	Blank cassette	1 per 20 cassette samples; minimum of 1 per batch	Not applicable—used to calculate spiked sample recovery
Spiked Sample, and duplicate spike cassette	1 mL of 100 μg/mL lead stock solution plus a blank cassette	1 per 20 cassette samples; minimum of 1 per batch	Accuracy of ±20% from average known value
Spiked Sample, and duplicate spike dust bottle	1 mL of 100 μg/mL lead stock solution into an empty beaker, equivalent to a spiked method blank	1 per 20 dust bottle samples; minimum of 1 per batch	Accuracy of ±20% from average known value

5.2 Document the condition of samples received in the laboratory notebook.

5.3 Label 100-mL Griffin beakers for each sample and associated quality control sample to be processed.

5.4 TRANSFER OF CASSETTE SAMPLES TO DIGESTION VESSELS

The cassette dust samples do not require weighing. Transfer each entire cassette dust sample into labeled Griffin beakers using the following procedure (see **NOTE 2**).

NOTE 2: For spikes and spike duplicates, transfer blank cassettes to beakers targeted for these samples and spike onto them according to details in Table K-1.

5.4.1 For cassette samples, carefully pry open and remove the top section of the filter cassette using a clean flat-edged screw driver, or equivalent tool. Place the top section upside down (in a manner which will not cause sample loss) on a clean Kimwipe™.

5.4.2 Gently empty the loose dust from the cassette into a labeled beaker. Care should be taken to prevent sample losses due to blowing or scattering of the dust.

5.4.3 Carefully rinse the inside of both the top section and remaining cassette assembly with 5 to 10 mL of 10% (v/v) HNO_3 transferring the rinse solution to the digestion beaker.

5.4.4 Carefully pry open and remove the retaining ring (placing it on a clean Kimwipe™) using a clean flat-edged screw driver, or equivalent tool.

5.4.5 Using acid cleaned polypropylene forceps carefully remove and transfer the filter to the digestion beaker.

5.4.6 Using the same forceps grasp the backup support pad and using the forceps and support pad, carefully swab the inside of the top section, retaining ring, and bottom section of the filter cassette. Then transfer the pad to the digestion beaker. This step is performed the help ensure that a quantitative sample transfer is achieved.

5.5 TRANSFER OF BOTTLED DUST SAMPLES TO DIGESTION VESSELS

The dust samples in bottles do not require weighing. Transfer each entire dust sample into labeled Griffin beakers using the following procedure (see **NOTE 3**). If the amount of dust contained in a bottle is too large, take a weighed aliquot for digestion.

NOTE 3: For spikes and spike duplicates, add 5 mL of 1:1 HNO_3 to each beaker and spike into empty beakers according to details in Table K-1.

5.5.1 Wipe off the outside of the bottle with a laboratory wipe (particularly the cap). Hold the top of the bottle over the beaker and carefully remove the top allowing any dust trapped in the cap to the fall into the beaker. While holding the cap over the beaker, rinse off the

inside surface into the beaker using about 1 to 2 mL of water from a squirt bottle filled with ASTM Type I water. Set the cap down (inside surface up) on a clean laboratory wipe.

5.5.2 Carefully tap the contents of the bottle into the beaker.

5.5.3 Place approximately 2 mL of 1:1 HNO_3 into the bottle, and recap. Gently turn the bottle to allow the solution to come into contact with all inside surfaces of the bottle. Uncap the bottle and pour the contents into the beaker.

5.5.4 Place approximately 2 mL of 1:1 HNO_3 into the bottle, and recap. Gently turn the bottle to allow the solution to come into contact with all inside surfaces of the bottle. Uncap the bottle and pour the contents into the beaker. (This is a repeat of step 5.5.3).

5.5.5 Place approximately 2 mL of 1:1 HNO_3 into the bottle, and recap. Gently turn the bottle to allow the solution to come into contact with all inside surfaces of the bottle. Uncap the bottle and pour the contents into the beaker. (This is a second repeat of step 5.5.3 for a total of three rinses of the bottle).

5.6 DIGESTION OF SAMPLES

Perform digestion of each sample as follows (see **NOTE 4**).

NOTE 4: For dust samples from bottles, skip to step 5.6.2 since acid was already used to quantitatively transfer the sample from the bottle to the beaker.

5.6.1 Add 5 mL of 1:1 HNO_3 to each beaker.

5.6.2 Gently swirl to mix, and cover with a watch glass. Gently heat the sample to 85° to 100°C and reflux for 10 to 15 min without boiling.

5.6.2 Allow the sample to cool.

5.6.3 Add 2.5 mL of concentrated HNO_3, cover with watch glass and reflux for 30 min without boiling.

5.6.4 Repeat this last step (5.6.3) to ensure complete oxidation.

5.6.5 Remove watch glass and allow the solution to evaporate to approximately 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker (see **NOTE 5**).

NOTE 5: Because ribbed watch glasses are not available, the watch glasses may be removed to accelerate volume reduction while assuring minimal contamination of the samples through careful observation. When removing these watch glasses, care must be exercised to avoid losses by rinsing them with a minimum amount of Type I water (rinsed into the sample beaker) and avoiding contaminating them by placing them upside down on new clean Kimwipe™.

5.6.6 Allow the sample to cool.

5.6.7 Add 2 mL Type I water and 2 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

5.6.8 Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged (see **NOTE 6**).

NOTE 6: Do not add more than a total of 5 mL of 30% H_2O_2 even if effervescence has not been reduced to a minimal level.

5.6.9 Remove the watch glass and continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 2.5 mL (see **NOTE 5**).

5.6.10 Allow the digestates to cool, rinse the beaker walls and bottom of the watch glass into the digestate, and quantitatively transfer to a 25-mL volumetric flask. Dilute to volume with Type I water.

5.7 Particulates in the digestate should then be removed by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement. A disposable syringe equipped with an Acrodisc filter can be used to filter a portion of the sample digest prior to analysis.

5.8 The diluted digestate solution contains approximately 10% (v/v) HNO_3. Calibration standards used for instrumental measurement should be made with this level of HNO_3.

APPENDIX L

ANALYTICAL PROCEDURE

MODIFIED METHOD 6010A FOR THE ANALYSIS OF DIGESTED SAMPLES FOR LEAD (Pb) BY INDUCTIVELY COUPLED PLASMA (ICP-AES) PLUS MODIFIED METHODS FOR FLAME ATOMIC ABSORPTION (FAAS) AND GRAPHITE FURNACE ATOMIC ABSORPTION (GFAAS) TECHNIQUES

MODIFIED METHOD 6010A FOR THE ANALYSIS OF DIGESTED SAMPLES FOR LEAD (Pb) BY INDUCTIVELY COUPLED PLASMA (ICP-AES) PLUS MODIFIED METHODS FOR FLAME ATOMIC ABSORPTION (FAAS) AND GRAPHITE FURNACE ATOMIC ABSORPTION (GFAAS) TECHNIQUES

1.0 SUMMARY

A sample digestate is analyzed for lead content using ICP-AES, Flame-AAS, or Graphite Furnace-AAS techniques. Instrumental Quality Control samples are analyzed along with sample digestates to assure adequate instrumental performance. This procedure is a modification of the SW-846 Method 6010A.

2.0 DEFINITIONS

2.1 *Digestion* - The sample preparation process that will solubilize targeted analytes present in the sample and results in an acidified aqueous solution called the digestate.

2.2 *Digestate* - An acidified aqueous solution that results from performing sample preparation (digestion) activities. Lead measurements are made using this solution.

2.3 *Batch* A group of QC samples that are processed together using the same reagents and equipment.

2.4 *Serial Dilution* - A method of producing a less concentrated solution through one or more consecutive dilution steps. The dilution step for a standard or sample is performed by volumetrically placing a small aliquot of a higher concentrated solution into a volumetric flask and diluting to volume with water containing the same acid levels as found in original sample digestates.

2.5 *Method Blank* - A digestate that reflects the maximum treatment given any one sample within a sample batch except that it has no sample initially placed into the digestion vessel. (The same reagents and processing conditions that are applied to samples within a batch also are applied to the method blank.)

Analytical results from method blanks provide information on the level of potential contamination experienced by samples processed within the batch.

2.6 *No-Spiked Sample* A portion of a homogenized sample that is targeted for addition of analyte but that was not fortified with all the target analytes before sample preparation. A method blank serves as a no-spike sample in cases where samples can not be uniformly split, as described in section 2.7. Analysis results for this sample are used to correct for native analyte levels in the spiked and spiked duplicate samples.

2.7 *Spiked Sample and Spiked Duplicate Sample* - Two portions of a homogenized sample that are targeted for addition of analyte and are fortified with all the target analytes before preparation. In cases where samples can not be uniformly split (such as paint chip samples taken for lead per area determinations), a method blank can be used in place of the homogenized sample split. Use of a method blank for a spiked sample should be referred to as a "spiked method blank" or "spiked method blank duplicate." Analysis results for these samples are used to provide information on accuracy and precision of the overall analysis process.

2.8 *Analysis Run* - A period of measurement time on a given instrument during which data is calculated from a single calibration curve (or single set of curves). Re-calibration of a given instrument produces a new analysis run.

2.9 *Instrumental QC Standards* - Solutions analyzed during an instrumental analysis run that provide information on measurement performance during the instrumental analysis portion of the overall lead measurement process.

2.10 *Semi-quantitative Screen* - An analysis run that is performed on highly diluted sample digestates for the purpose of determining the approximate analyte level in the digest. This analysis run is generally performed without inserting Instrumental QC standards except for calibration standards. Data from this run are used for determining serial dilution requirements for sample digestates to keep them within the linear range of the instrument.

2.11 *Quantitative Analysis* - An analysis run on sample digestates (or serial dilutions of sample digestates) that includes Instrumental QC standards. Data from this run are used to calculate and report final lead analysis results.

2.12 *Initial Calibration Blank (ICB)* A standard solution which contains no analyte and is used for initial calibration and zeroing instrument response. The ICB must be matrix matched to acid content present in sample digestates. The ICB must be measured during calibration and after calibration. The measured value is to be less than 5 times the instrumental detection limit.

2.13 *Calibration Standards* Standard solutions used to calibrate instrument. Calibration standards must be matrix matched to acid content present in sample digestates and must be measured prior to measuring any sample digestates.

2.14 *Initial Calibration Verification (ICV)* A standard solution (or set of solutions) used to verify calibration standard levels. Concentration of analyte near mid-range of linear curve that is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. The ICV must be matrix matched to acid content present in sample digestates. The ICV must be measured after calibration and before measuring any sample digestates. The measured value to fall within $\pm$10% of known value.

2.15 *Interference Check Standard (ICS)* - A standard solution (or set of solutions) used for ICP-AES to verify accurate analyte response in the presence of possible spectral interferences from other analytes present in samples. The concentration of analyte is to be less than 25% of the highest calibration standard, concentrations of interferant will be 200 μg/Ml of Al, Ca, Fe, and Mg. The ICS must be matrix matched to acid content present in sample digestates. The ICS must be analyzed at least twice, once before and once after all sample digestates. The measured analyte value is expected to be within $\pm$20% of known value.

2.16 *Continuing Calibration Verification (CCV)* - A standard solution (or set of solutions) used to verify freedom from excessive instrumental drift. The concentration is to be near mid-range of linear curve. The CCV must be matrix matched to acid content present in sample digestates. The CCV must be analyzed before and after all sample digestates and at a frequency not less than every 10 sample digestates. The measured value to fall within $\pm$10% of known value for ICP-AES or FAAS ($\pm$20% for GFAA), run once for every 10 samples.

2.17 *Continuing Calibration Blank (CCB)* - A standard solution that has no analyte and is used to verify blank response and freedom from carryover. The CCB

must be analyzed after the CCV and after the ICS. The measured value is to be less than 5 times the instrumental detection limit.

3.0 APPARATUS AND MATERIALS

3.1 Analytical Instrumentation

3.1.1 Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES). The ICP-AES, either sequential or simultaneous, must be capable of measuring at least one of the primary ICP lead emission lines. Emission line used must be demonstrated to have freedom from common major interferants such as Al, Ca, Fe and Mg or the ability to correct for these interferants.

3.1.2 Flame Atomic Absorption Spectrometer (FAAS) - Equipped with an air-acetylene burner head, lead hollow cathode lamp or equivalent and capable of making lead absorption measurements at the 283.3-nm absorption line.

NOTE: The 283.3-nm line is preferred over the 217-nm line because of the increased noise levels commonly observed at the 217-nm line for FAAS and GFAAS.

3.1.3 Graphite Furnace Atomic Absorption Spectrometer (GFAAS) Equipped with background correction and lead hollow cathode lamp or equivalent and capable of making lead absorption measurements at the 283.3-nm absorption line.

3.2 Gases

Grades specified by manufacturer of the instrument employed.

3.2.1 Compressed air and acetylene for FAAS.

3.2.2 Compressed or liquid argon for ICP-AES and GFAAS.

3.2.3 Minimum of two stage regulation of all gases.

3.3 Vinyl Gloves: Powderless

3.4 Micropipettors with Disposable Plastic Tips

Sizes needed to make reagent additions, and spiking standards. In general, the following sizes should be readily available: 1-5 mL adjustable, 1000 µL, 500 µL, 250 µL, and 100 µL.

3.5 Volumetric Flasks

Sizes needed to make calibration standards, serial dilutions, and Instrumental QC standards.

4.0 REAGENTS

4.1 *Nitric acid*, concentrated and reagent grade

4.2 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 of Specification D1193. (ASTM Type I water: Minimum resistance of 16.67 megohm-cm, or equivalent.)

4.3 *Calibration stock solution*, 100 µg/mL of Pb in dilute nitric acid or equivalent (such as a multielement stock containing Pb).

4.4 *Check standard stock solution* (for ICV), 100 µg/mL of Pb in dilute nitric acid or equivalent. Must be sourced from a different lot number (or manufacturer) than the calibration stock solution (7.3).

4.5 *Interferant stock solution* (for ICS; ICP-AES only), 10000 µg/mL of Al, Ca, Fe, and Mg in dilute nitric acid or equivalent.

5.0 PROCEDURE

5.1 *Laboratory Records*—Record all reagent sources (lot numbers) used for sample preparation in a laboratory notebook. Record any inadvertent deviations, unusual happenings, or observations on a real-time basis as samples are processed. Use these records to add supplement lead data when reporting results.

5.2 *Instrumental Setup*

5.2.1 *FAAS/GFAAS* - Set the FAAS or GFAAS spectrometer up for the analysis of lead at 283.3 nm according to the instructions given by the manufacturer. Be sure to allow at least a 30-min warmup of the hollow cathode lamp prior to starting calibration and analysis.

5.2.2 *ICP-AES* Set the ICP spectrometer up for the analysis of lead at a primary lead emission line (such as 220.2 nm) according to the instructions given by the manufacturer. Be sure to allow at least a 30-min warmup of the system prior to starting calibration and analysis.

5.3 *Preparation of Calibration and Instrumental QC Standards*

5.3.1 *Calibration Standards* - Prepare a series of calibration standards covering the linear range of the instrumentation. Prepare these standards using serial dilution from the calibration stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates. Also prepare an Initial Calibration Blank (ICB) as defined in Section 3 and Table L-1.

NOTE: For FAAS/GFAAS prepare a minimum of 3 calibration standards plus the ICB for performing calibration of the instrument. ICP-AES can be performed using one high calibration standard and an ICB. However, more are generally preferred.

5.3.2 *Instrumental QC Standards* Prepare Instrumental QC standards as summarized in Table L-1 using serial dilution from the required stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates.

NOTE: The ICV is used to assess the accuracy of the calibration standards. Therefore, it must be made from a different original source of stock solution than the stock used to make the calibration standards. Use of a different serial dilution of the same original stock is not acceptable.

5.4 *Calibration and Instrumental Measurement* Perform calibration and quantitative lead measurement of sample digestates and instrumental QC samples in the sequential order outlined in Table L-2.

NOTE: Performance of a semi-quantitative screen prior to quantitative analysis for sample digests containing unknown levels of lead is generally recommended. The purpose of this screen is to determine serial dilution requirements of each digestate needed to keep the instrumental response within the calibration curve. During a semi-quantitative screen, all digestates are diluted to a constant large value (1-to-100 for ICP/FAAS and 1-to-1000 for GFAAS). The instrument is calibrated and diluted digestates are analyzed without inserting the instrumental QC used for a quantitative analysis run. Data from this screen are reviewed to calculate the optimum serial dilution needed for each digestate. <u>No sample data can be reported for any analyte value not falling within the calibration range</u>. Therefore, the optimum dilution is one that achieves the maximum lead response still within the calibration curve. For ICP-AES, levels of possible interferants (Al, Ca, Fe and Mg) also may have to be considered in order to make interference corrections. For ICP-AES, digestates must be sufficiently diluted to assure that levels of possible interferants, such as Al, Ca, Fe and Mg, are at or below the levels present in the ICS.

5.5 *Instrumental QC Evaluation and Corrective Action* Examine the data generated from the analysis of calibration standards and Instrumental QC standards. Evaluate the analysis run using the criteria shown in Table L-1. Failure to achieve the specifications shown in Table L-1 will require corrective action to be performed as described below:

5.5.1 *ICB, Calibration standards, or ICV* - Failure to meet specifications for these Instrumental QC standards requires complete re-calibration. Sample digestates can not be measured under these conditions. It is recommended that standards be re-prepared prior to re-calibration.

5.5.2 *High Calibration Standard Re-run* - Failure to meet specifications for this Instrumental QC standard requires complete re-calibration. Sample digestates can not be measured under these conditions. It is recommended that standard range be reduced prior to re-calibration.

5.5.3 *ICS* - Failure to meet specifications for these Instrumental QC standards requires re-analysis of the standard until specifications are met. Sample digestates can not be measured under these conditions. Re-preparation of the standard prior to re-analysis is recommended under these conditions. Continued failure of the ICS may require interference correction investigation or changing of instrument parameters.

Consult the manufacturer's recommendations under these conditions. Any change in instrument parameters must be accompanied by recalibration. If measured aliquots of sample digestates can be shown to contain interferants less than those recommended for the ICS, then the interference levels in the ICS can be lowered. Such changes must be documented in laboratory records with data supporting the justification for the change. All measurements on sample digests must be bracketed by an ICS that meets specifications (called a "passing" ICS). Failure to meet specifications on the ICS run after the sample digestates requires re-running all sample digestates since the last passing ICS was measured. Since only the ICS is required to be analyzed twice, much data could be lost if the analytical run were long and the second ICS failed specifications. This is a good reason for including periodic analysis of the ICS as shown in Table L-2.

5.5.4 *CCV* - Failure to meet specifications for these Instrumental QC standards indicates excessive instrumental drift. Sample digestates can not be measured under these conditions and any sample digestates measured since the last passing CCV must be reanalyzed. This situation requires either re-analysis of the standard until specifications are met or re-calibration. All measurements on sample digests must be bracketed by an CCV which meets specifications.

5.5.5 *CCB* - Failure to meet specifications for these Instrumental QC standards indicates the presence of possible instrumental carryover or baseline shift. Such a failure will have the most impact on sample digestates at the lower end of the calibration curve. The first corrective action is to re-analyze the CCB. If the CCB passes, then the rinse time between the samples should be increased and the analysis continued. If the instrument response is still elevated and has not significantly changed, then the instrument can be re-zeroed followed by a CCV-CCB and re-analysis of all samples since the last passing CCB that are within 5 times the response of the failed CCB.

6.0 CALCULATIONS

For FAAS/GFAAS : Prepare a calibration curve to convert instrument response (absorbance) to concentration (μg/mL) using a linear regression fit. Convert all instrumental measurements on instrumental QC standards and sample digests to lead concentration (μg/mL) using the calibration curve.

NOTE: Some instruments will automatically prepare a calibration curve based on a linear regression fit.

For ICP-AES: All modern ICPs automatically prepare a calibration curve to convert instrumental responses (emission intensity) to concentration (μg/ml).

Based on the volume of the solution analyzed (and considering any necessary dilutions), use the concentration results to calculate the total μg of Pb in the original sample that was digested. If the original sample was dust in a bottle and the sample amount was too large so that a weighed aliquot was taken, divide the analysis result (μg) by the weight of the aliquot and report the result as μg/g of dust.

Table L-1. SUMMARY OF LABORATORY INSTRUMENTAL MEASUREMENT QC STANDARDS

Name	Use	Specification	Frequency
ICB - Initial calibration blank	Used for initial calibration and zeroing instrument response.	Calibration standard which contains no analyte. Measured value to be less than 5 times the instrumental detection limit.	Must be measured during calibration and after calibration.
Calibration standards	Used to calibrate instrument. The high standard rerun is used to check for high response rollover.	Must be matrix matched to acid content present in sample digestates. Correlation coefficient of $\geq$0.995, as measured using linear regression on instrument response (y) versus concentration (x). Measured value of highest level calibration standard to fall within $\pm$10% of known value.	Must be measured prior to measuring any sample digestates. The highest level calibration standard must be measured after calibration.
ICV - Initial calibration verification	Used to verify calibration standard levels.	Concentration of analyte to be near mid-range of linear curve, which is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. Measured value to fall within $\pm$10% of known value.	Must be measured after calibration and before measuring any sample digestates.

Table L-1. SUMMARY OF LABORATORY INSTRUMENTAL MEASUREMENT QC STANDARDS (continued)

Name	Use	Specification	Frequency
ICS - Interference check standard (for ICP-AES only)	Used to verify accurate analyte response in the presence of possible spectral interferences from other analytes present in samples.	Concentration of analyte to be less than 25% of the highest calibration standard, concentrations of interferant will be 200 μg/mL of Al, Ca, Fe, and Mg. Measured analyte value to fall within ±20% of known value.	Must be analyzed at least twice, once before and once after all sample digestates.
CCV - Continuing calibration Verification	Used to verify freedom from excessive instrumental drift.	Concentration to be near mid-range of linear curve. Measured value to fall within ±10% of known value for ICP-AES or FAAS (±20% for GFAA).	Must be analyzed before and after all sample digestates and at a frequency not less than every 10 sample digestates.
CCB - Continuing calibration blank	Used to verify blank response and freedom from carryover.	Calibration standard which contains no analyte. Measured value to be less than 5 times the instrumental detection limit.	Must be analyzed after the CCV and after the ICS.

TABLE L-2. EXAMPLE OF A TYPICAL ANALYSIS ORDER FOR MEASUREMENT			
Run Order No. (relative)	**Sample ID**	**Comments**	
1	ICB	Calibration blank	Instrument calibration
2-4	low, med, high	Calibration standards	
5	ICB	Calibration blank	Calibration verification
6	ICV	Made from different stock, level is near mid-point of curve	
7	high standard	Calibration standard	Linearity check
8	CCB	Same as calibration blank	
9	ICS	Interference check standard	Interferant check for ICP only
10	CCB	Carryover check	
11	CCV	Drift check, same as near midpoint calibration standard	Continuing calibration verification
12	CCB	Carryover check	
*** start repeating cycle of samples-Instrumental QC here ***			
13-22	Sample IDs	Sample digestates	Max. of 10 samples
23-24	CCV CCB	Drift check + Carryover check	See run # 11-12
25-34	Sample IDs	Sample digestates	Max. of 10 samples
35-36	ICS CCB	Interferant check + Carryover check	See run # 9-10
37-38	CCV CCB	Drift check + Carryover check	See run # 11-12
*** end repeating cycle of samples-QC standards here ***			

APPENDIX M

GLASSWARE/PLASTICWARE CLEANING PROCEDURE

ATOMIC SPECTROMETRY FACILITY STANDARD OPERATING PROCEDURE	Code: ASF-201 Revision: 1 Date: 02/9/93 Page: 1 of 8

Title: Glassware/Plasticware Cleaning Procedure

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Approved: John D. Wett for Dir. Gray
Chemical Sciences Department Director

Eugene G. Podrebarac
Quality Assurance Unit

1.0 SUMMARY

This standard operating procedure is used to clean laboratory glassware and plasticware (labware) in the Atomic Spectrometry Facility's Sample Preparation Laboratory. Labware is cleaned with detergent and nitric acid solution. Labware prepared by this procedure can be used for field sample collection and routine sample preparation and analysis work.

The following items are exempt from this procedure because screening analyses have been performed and these items do not contribute to positive method blanks: disposable pipette tips, disposable polystyrene beakers, disposable autosampler tubes/cups, centrifuge tubes, and disposable Falcon tubes. Additional items may also be exempt from this procedure if screening analysis determines that these items are free from contamination of the target analytes, at the levels of interest.

2.0 REQUIRED PRELIMINARY INFORMATION

The following information must be provided before this procedure may be conducted:

- Charge number.
- Total amount of glassware to be processed.
- Material Safety Data Sheet for Nitric Acid (HNO_3). This must be read before using nitric acid.

3.0 APPARATUS AND MATERIALS

3.1 Glassware tubs labeled as: For Clean Glassware and only used for clean glassware.

3.2 Glassware tubs labeled as: For Dirty Glassware and only used for dirty glassware.

3.3 Pipette cylinder: for DI water rinsing

3.4 Pipette cylinder: for Milli-Q water rinsing

3.5 Pipette cylinder: for acid cleaning

3.6 Pipette basket: for acid cleaning and rinsing

3.7 Large acid tank: for acid cleaning

3.8 Brushes: plastic bristle, assorted sizes

3.9 Face shield

3.10 Gloves: heavy duty neoprene or nitrile (green), acid resistant

3.11 Gloves: disposable, powderless, vinyl or latex

3.12 Acid resistant protective clothing: rubber or poly-laminated tyvek apron

3.13 Filtering flask

3.14 Aspirator pump

3.15 Labware drainer

3.16 Pipette drying racks

3.17 Labware drying racks

3.18 Paper towels

3.19 Food service towels, or equivalent

3.20 Plastic bags: assorted sizes, for storage of clean, dry pipettes and labware

3.21 Plastic squirt bottle: 500 mL, for baking soda slurry

4.0 REAGENTS

4.1 Acetone: ACS reagent grade, or equivalent

4.2 Deionized (DI) water

4.3 Alconox detergent

4.4 Acationox detergent

4.5 Concentrated nitric acid (HNO_3): ACS reagent grade

4.6 Approximately 20% to 30% (v/v) nitric acid (HNO_3): for acid cleaning (prepare by adding 1 part concentrated HNO_3 to 3 parts DI water, as described in AS-201, Acid Bath Maintenance Procedure)

4.7 Milli-Q water (Type I Water): 16.67 megohm-cm minimum resistance, or equivalent

4.8 Baking soda (sodium bicarbonate): for large acid spills

4.9 Baking soda slurry: for small acid spills (prepare by adding about 50 g baking soda to a 500-mL plastic squirt bottle, then diluting to volume with DI water)

5.0 SAFETY REQUIREMENTS

The use of large amounts fo nitric acid requires vigilance to avoid splashes and spills. Before utilizing this procedure, an individual must observe and be instructed in the proper technique for using the acid bath.

5.1 A Class A fume hood, verified to be in operating condition, must be used for the acid bath solutions.

5.2 Safety glasses with side shields must be worn at all times in the laboratory. A face shield should be used when handling large pieces of glassware (e.g., 1-L beakers).

5.3 Neoprene gloves with vinyl or latex glove liners are required when working with the acid bath or transferring large volumes of acid solutions.

5.4 Lab coats are required for working with the acid bath. Acid-resistant protective clothing, such as a rubber apron, is also recommended.

5.5 An extra person must be present in the laboratory area when concentrated acid transfers are being performed, in order to help handle problems in case of inadvertent spills or exposure.

5.6 The closest shower and eye wash station must be noted before beginning this procedure. It is located immediately before the washing area.

5.7 A squirt bottle containing the baking soda (sodium bicarbonate) slurry and an open box of baking soda must be in the work area for neutralizing any spills. Use the slurry for neutralizing small acid spills, including those on clothing. Use the bulk material for neutralizing larger spills.

5.8 When mixing acid solutions, **ALWAYS** add the acid to the water to prevent potentially violent reactions. **NEVER** add water to acid.

6.0 QUALITY CONTROL

The acid cleaning tanks containing approximately 20% to 30% (v/v) HNO_3 are routinely monitored for contamination as described in ASF-202, Acid Bath Maintenance Procedure.

7.0 GLASS VOLUMETRIC PIPETTE CLEANING PROCEDURE

NOTE: Aspiration is conducted using an aspirator pump attached to a filtering flask. Do not aspirate from the delivery tip of a pipette.

7.1 **Preclean with Alconox solution if pipettes are contaminated with organic compounds.** Pipettes are contaminated with organic compounds when used with solid organic waste, municipal wastewater, etc. If organic contamination is in doubt, ask the project leader.

7.1.1 Scrub the outside of the pipettes using Alconox solution and a food service towel.

7.1.2 Aspirate 2 pipette volumes of Alconox solution (prepared with DI water according to container's instructions) through each pipette.

7.1.3 Rinse off the outside of the pipette with DI water until suds are gone.

7.1.4 Aspirate DI water through each pipette until the suds are gone.

7.2 **Clean with Acationox solution.**

7.2.1 Aspirate 2 aliquots Acationox solution (prepared with DI water according to container's instructions) through each pipette.

7.2.2 Aspirate DI water through each pipette until the suds are gone.

7.2.3 Rinse the outside of the pipettes with DI water.

7.3 **Clean with nitric acid solution.**

7.3.1 Place pipettes tip-up in the pipette basket. If the basket is not already in the acid cylinder containing approximately 20% to 30% (v/v) HNO_3, carefully transfer the basket to the cylinder.

7.3.2 Allow pipettes to soak for at least 15 min.

7.3.3 Slowly lift the basket, hook basket on hook inside hood, and allow the acid solution to drain back into the acid bath.

7.3.4 Transfer pipettes in the basket into the pipette DI rinsing tank. If needed, connect the DI waterline to the rinsing tank. Turn on the DI water. Allow the DI water to flow into the rinsing tank until a minimum of 2 tank volumes have passed through the tank. Lift the basket and allow any excess DI water to drain back into the rinsing tank.

7.3.5 Transfer pipettes in the basket to a Milli-Q cylinder. If the cylinder is not already filled, fill with Milli-Q water. Slowly lift the basket and allow the water to drain from the pipettes back into the bath. Submerge the basket and pipettes into the cylinder, allow the pipettes to fill with water. Lift the basket and drain the pipettes again.

7.3.6 Carefully and slowly transfer pipettes to a <u>Clean Glassware</u> tub for movement to a clean drying area or transfer directly into a glassware cart drying rack lined with food towels.

7.3.7 If a clean glassware tub was used for transfer, stack pipettes tip-up in a pipette drying rack or glassware cart drying rack and cover with a clean food service towel.

7.3.8 Allow pipettes to dry completely at ambient temperature.

7.3.9 Store the clean dry pipettes in a clean drying area until use.

8.0 GENERAL LABWARE CLEANING PROCEDURE

8.1 Prepare labware for cleaning.

8.1.1 Place dirty labware in <u>Dirty Glassware</u> tubs.

8.1.2 Remove all identifying marks from the labware such as labeling, barcodes, and tape. Remove indelible marking pen labels by wiping with a paper towel saturated with acetone (perform inside or immediately in front of a fume hood).

CAUTION: Acetone will react with and dissolve vinyl gloves, therefore wear latex or nitrile gloves when using acetone. In addition, acetone may react with some plasticware.

8.2 Preclean with Alconox solution if labware is contaminated with organics.

8.2.1 Fill a Dirty Glassware tub with Alconox solution (prepared with hot tap water according to container's instructions).

8.2.2 Allow labware to soak for a sufficient amount of time (approximately 2 to 4 hr) to remove organic residues from the surface of the labware (see **NOTE 1**).

NOTE 1: CAUTION: Do not soak for more than 8 hr. Alconox will remove the white permanent label areas from glassware.

8.2.3 Scrub labware inside and out with an appropriate plastic bristle brush or food service towel until they are clean.

8.2.4 Rinse labware inside and out a minimum of 3 times with DI water, then drain. Label if not proceeding as Labware Ready for Acationox Cleaning.

8.3 Clean with Acationox solution.

8.3.1 Fill a Dirty Glassware tub with Acationox solution (prepared with DI water according to directions on the container).

8.3.2 Transfer labware to the tub. Scrub labware inside and out with an appropriate plastic bristle brush or food service towel until they are clean. Some labware, such as volumetric flasks, cannot be easily cleaned inside using a brush or towel. For these types of labware vigorously shake an aliquot of Acationox solution within the vessel to help with cleaning.

8.3.3 Rinse labware inside and out 3 or 4 times with DI water.

8.3.4 Allow labware to drain for several minutes, by inverting them in a drainer marked with a sign that identifies them as "ready for acid bath." In general, use of drainers in glassware carts is recommended.

8.3.5 If labware will not be immediately cleaned in the acid bath, cover labware with food service towels (to prevent contamination from airborne dust) and label as Labware Ready for Acid Bath.

8.4 Clean with nitric acid solution.

8.4.1 Carefully submerge labware into the acid bath tanks containing approximately 20% to 30% (v/v) HNO_3.

8.4.2 Allow labware to soak at least 15 min.

CAUTION: Avoid soaking plasticware longer than 8 h (to prevent damage from long term exposure to the acid solution).

8.4.3 Carefully remove labware from the tank, drain any acid back into the tank (see **NOTE 2**).

NOTE 2: If gloves come into contact with acid, minimize spillage caused by acid drips from gloves by limiting movement of gloves outside the hood. One method is the use of a separate plastic tub or other equivalent container inside the hood for placement of acid containing gloves. Other methods which will minimize spillage can be used. If spillage occurs, neutralize with baking soda as described in Section 5.7.

8.4.4 If the labware is to be rinsed within the acid bath hood using a rinse tank, then transfer labware to a clean glassware tub containing Milli-Q water to transfer the glassware, then rinse labware a minimum of 3 times with Milli-Q water.

If the labware is to be rinsed outside of the acid bath hood, then transfer the labware to a clean glassware tub containing Milli-Q water to eliminate the presence of acid fumes. Rinse labware a minimum of 3 times with Milli-Q water.

8.4.5 Stack labware on drying racks lined with clean food service towels. Cover labware with additional clean food service towels (to prevent contamination from airborne dust). Label as Clean Labware and place into a clean drying area.

8.4.6 Allow labware to dry completely at ambient temperature.

8.4.7 Store labware in sealed plastic bags until use.

APPENDIX N

ACID BATH MAINTENANCE PROCEDURE

Title: Acid Bath Maintenance Procedure

Author: N. Friederich, G. Dewalt

Approved: John D. Witt for Don Gay
Chemical Sciences Department Director

Eugene E Podrebarac
Quality Assurance Unit

1.0 SUMMARY

Glassware and plasticware cleaning for the Atomic Spectrometry Facility is performed in the sample preparation laboratory, using acid cleaning baths containing approximately 20% to 30% (v/v) nitric acid (HNO_3). This standard operating procedure describes the set-up, routine screening (for metals contamination), and replenishing of pipette and general labware acid cleaning baths.

2.0 REQUIRED PRELIMINARY INFORMATION

The following information must be provided before this procedure may be conducted:

- Charge number.
- Material Safety Data Sheet for Nitric Acid (HNO_3) is available for review in the MSDS notebook, located in the ASF data reduction room.

3.0 ACID BATH MAINTENANCE LOGBOOK

All activities related to acid bath maintenance are recorded in a laboratory notebook dedicated to the acid bath. Logbook entries must be made each time a bath is filled, volume adjusted, or screened. It is the acid bath maintenance staff's responsibility to assure that the log is completed as required.

4.0 APPARATUS AND MATERIALS

4.1 Tygon tubing

4.2 Pipette cylinder: for acid bath cleaning

4.3 Large acid tank: for acid bath cleaning

4.4 Large acid tank: for neutralization

4.4 Large stirring rods

4.6 Large plastic drum: for acid waste disposal storage

4.7 Eppendorf pipettes with disposable tips

4.8 Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES)

4.9 Face shield

4.10 Gloves: heavy duty, neoprene or nitrile (green), acid resistant

4.11 Gloves: disposable, powderless, vinyl, and latex

4.12 Acid-resistant protective clothing: rubber or poly-laminated tyvek apron

4.13 Food service towels, or equivalent

4.14 Plastic squirt bottle: 400 mL, for baking soda slurry

4.15 Plastic baskets

4.16 Plastic hand pump suitable for nitric acid

5.0 REAGENTS

5.1 Deionized (DI) water: used to prepare the acid bath solution

5.2 Concentrated nitric acid (HNO_3): ACS reagent grade

5.3 Milli-Q water (Type I water): minimum resistance of 16.67 megohm-cm, or equivalent, used to dilute test samples for analysis

5.4 Baking soda (sodium bicarbonate): bulk (without any of the potential target metal analytes as a major contaminant (except sodium)

5.5 Baking soda slurry: prepare by adding about 50 mL baking soda (use a 50 mL disposal beaker) to a 500 mL plastic squirt bottle, then diluting to volume with DI water

5.6 Full range pH paper

5.7 Acationox detergent

6.0 SAFETY REQUIREMENTS

6.1 A Class A fume hood, verified to be in operating condition, must be used when preparing and using acid bath solutions.

6.2 Safety glasses are required for all work and a face shield is recommended. Face shields are required for any work involving transfers of concentrated acid or large volumes (> 4 L) of acid solutions.

6.3 Neoprene or nitrile gloves with vinyl or latex glove liners are required when working with the acid bath or transferring large volumes of acid solutions.

6.4 Labcoats are required for working with the acid bath. Acid-resistant protective clothing is required when transferring large volumes of acid solutions.

6.5 An extra person is required to be present in the laboratory area when concentrated acid transfers are being performed, in order to help handle problems in case of inadvertent spills or exposure.

6.6 The closest shower and eye wash station must be noted before beginning this procedure. They are located in front of the acid bath hoods.

6.7 A squirt bottle containing the baking soda slurry and an open box of baking soda must be in the work area to neutralize spills. The baking soda slurry is used to neutralize any small spills in the hood or on clothing. The baking soda in the box is used to neutralize larger spills.

6.8 When mixing acid solutions, **ALWAYS** add the acid to the water to prevent potentially violent reactions. **NEVER** add water to acid.

6.9 Avoid putting your head <u>and</u> face inside the hood.

7.0 ACID BATH PREPARATION PROCEDURE: TANKS AND PIPETTE CYLINDERS

7.1 Attach a piece of tygon tubing to the DI water tap and place the outlet in the bath.

7.2 Fill the acid bath tank with DI water to the DI water mark on the side of the bath (or cylinder).

7.3 Carefully add ACS reagent grade HNO_3 to the fill mark on the side of the tank (or cylinder).

7.4 Stir the clean acid bath mixture with a large stirring rod. NOTE: The resulting solution is approximately 20% to 30% (v/v) HNO_3.

7.5 Record the fill date in the Logbook.

7.6 Screen the contents as described in Section 8.0.

8.0 ACID BATH SCREENING PROCEDURE

Acid baths for glassware preparation are used for cleaning glassware until the concentration of metallic compounds in the baths rise above specified levels. The acceptable contamination levels, shown in Table 1, have been established based on experimental data. These levels are not expected to result in addition of significant levels of contaminates to samples prepared using glassware cleaned in the acid baths. This screening procedure must be performed when the bath is first set up, whenever it is replenished, approximately once per month with normal sample preparation and analysis activities, and on an as-needed basis when sample preparation and analysis activities are reduced.

A given acid bath is screened by performing ICP-AES analysis of a one to one dilution of an aliquot from the bath followed by comparison to Table 1 levels. The procedure is detailed below.

8.1 Remove a 3-mL to 5-mL aliquot from each acid bath.

8.2 Dilute each aliquot with an equal amount of Milli-Q water for analysis.

Table 1. ACCEPTABLE LEVELS OF SELECTED ANALYTES IN ACID BATHS

Analytes	Acceptable level
Hg	< 10 µg/mL
Cd, Se, Ag, As, Ba, Cr, Pb, Mn, Sn Zn	< 25 µg/mL
Al, Ca, Fe, and Mg	< 100 µg/mL

8.3 Analyze the above dilution for the analytes shown in Table 1 by ICP-AES. NOTE: The acid concentration of the test sample is now about approximately 10% to 15% (v/v) HNO_3.

8.4 Multiply the analysis results by a dilution factor of 2.

8.5 Record the analytical results in the Logbook.

8.6 Compare the results to the criteria shown above in Table 1.

8.7 If the concentration of any analyte is greater than its acceptable level, label the acid bath: DO NOT USE—ACID MUST BE CHANGED, discard the bath contents, clean the bath and lids as described in Section 9.0, and replenish as described in Section 7.0. If the concentrations are close to but still beneath the acceptable level, contact the facility manager.

8.8 If the results are exceedingly high, contact the facility manager and investigate the probable cause, documenting the results of the investigation in the Logbook.

8.9 If the results are within the acceptable levels and the total volume levels in the bath are too low for use, add three parts DI water and one part concentrated HNO_3 until the acid baths are filled to the original fill mark. Document the addition in the Logbook.

9.0 DISPOSAL

Acid baths which fail to meet acceptable contaminate levels must be emptied, rinsed and refilled. The acid bath contents are transferred to a plastic drum identified for acid waste prior to disposal. Two options—Indirect Pumping and Siphoning—are available for bulk transfer of the acid to the drum. Direct Pumping is only used for transferring the acid tank rinsate to the holding tank. Siphoning is presented as an option; however it

should be used only when Direct and Indirect Pumping are not possible. Handling large quantities of acid require the use of a face shield and acid resistant protective clothing.

9.1 Direct Pumping

Direct pumping of liquid from acid baths is the process of using a plastic hand pump to transfer the liquid from the bath directly into the plastic drum identified for acid waste (referred to as "Acid Waste Bulk" drum). A hand pump is used as opposed to a mechanized pump to increase the operator control of this transfer and reduce the chances of a spill involving large volumes. Direct pumping should be used only for transfer of liquid out of the narrow acid waste tanks located in the ASF acid bath hoods. This is to reduce the risks of inadvertent contamination of a given acid bath system caused by insertion of a contaminated plastic hand pump. However, if required, direct pumping can be used to remove acid from any acid bath. Under such circumstances, the hand pump should be thoroughly cleaned and rinsed prior to use. Perform direct pumping as listed below.

9.1.1 Remove the plastic pump from storage and check the pump shaft for cleanliness using visual inspection. Rinse off any observed materials which may result in further contamination of the bath using DI water.

9.1.2 Place "Acid Waste Bulk" drum within easy reach of the pump tubing. If the drum is not already connected to venting, remove the venting bung and secure a venting tube to the drum.

9.1.3 Remove the fill bung of the "Acid Waste Bulk" drum and secure the plastic flex hose from the plastic pump to the drum. Place the plastic pump shaft into the tank targeted for emptying and pump the liquid waste into the drum (see NOTE 1).

> NOTE 1: The "Acid Waste Bulk" drum must not be filled completely full to allow for expansion and contraction during transport to the disposal site. Prior to pumping acid into the drum, examine the drum for sufficient space to accommodate the contents of the acid bath. A dip stick can be used to check the drum volume level. The "Acid Waste Bulk" drum final volume should not exceed 90% of the total drum volume.

9.1.4 Upon completion of transfer, pump several volumes of air to assure no significant volumes of liquid remain in the pump. Remove the plastic flex hose from the "Acid Waste Bulk" drum and replace the fill bung. Remove the plastic pump from the acid tank, rinse it, place it

in a plastic bag or other suitable plastic container, and store for later use.

9.1.5 If desired, remove the venting tube from the "Acid Waste Bulk" drum and replace the bung. Store the "Acid Waste Bulk" drum out of the way in a area with good ventilation.

9.2 Indirect Pumping

Indirect pumping is the process of using the air-driven pumping system built into the acid bath hoods to transfer liquids from the acid baths into the narrow acid waste tank mounted in the hoods. Perform indirect pumping as described below.

9.2.1 Verify that the pump source piping or tubing is placed into the acid tank targeted for transfer. If it is not, then affix the pipe or tubing as needed.

9.2.2 Identify which acid tank number is to be emptied (1, 2, or 3). Use this number to identify the correct "source valve" located under the diaphragm pump assembly on the side on the hood.

9.2.3 Open the "water feed" valve for approximately 3 sec to initiate priming of the diaphragm pump then shut it off.

9.2.4 Open the "source valve" corresponding to the acid tank targeted for transfer.

9.2.5 Open the "on/off" pump valve (air feed) to start pumping (See Notes 2 and 3).

Open the "water feed" valve again for approximately 3 sec to complete priming of the diaphragm pump (while the pump is running) then shut it off.

Monitor the pumping of the acid bath contents into the narrow acid tank. Take care not to overflow the narrow acid tank by stopping the pump when the volume reaches about 3/4 of the total tank volume. Stop the pumping by closing the "on/off" pump valve (air feed).

NOTE 2: Check the air pressure feeding the "on/off" pump valve when the pump is running. It should be set to between 10 psi and 25 psi. It is best to operate the pump as slow as possible to reduce splashing: the lower the pressure, the

slower the pumping rate. If the pressure is too low, the pump will not function.

NOTE 3: If the pump does not operate and the pressure appears to be sufficiently high, then close the "on/off" pump valve (air feed), push in the reset button located on the diaphragm pump, and restart the procedure from the beginning of Section 9.2.5.

9.2.6 Upon completion of the transfer of liquid from the acid baths to the narrow acid tank, close the "source valve" corresponding to the acid bath targeted for transfer.

9.3 Siphoning

Siphoning is the process of transferring acid bath contents using a hose filled with liquid. This method should only be used when direct or indirect pumping is not possible. Extra care must be taken when using this method for liquid transfer to avoid exposure to acid solutions and fumes. Any siphoning of acid bath contents must be performed with two people to help reduce the hazards of this type of acid transfer.

An ~ 1/2-in inner diameter (i.d.) rubber hose of sufficient length is filled with water, keeping both ends fo the hose at the same level. Close one end with a clamp (while both ends are level) and place the other end in the bath. Place the end with the clamp in the narrow acid tank and open the clamp.

10.0 CLEANING

10.1 Previously used Acid Baths

Acid baths which have been emptied of acidic contents are cleaned prior to refilling by rinsing with DI water as described below.

10.1.1 Using a clean food service towel dampened with DI water, wipe off the top surface of the acid bath lid (if present) to remove any foreign materials. Connect a clean tygon hose to the DI water tap and spray down the inside walls of the tank lid into the tank. Rinse a minimum of 3 times using sufficient DI water to cover the entire surface area of the lid.

10.1.2 Using the clean tygon hose connected to the DI water tap, thoroughly rinse the inside walls of the tank. Use sufficient DI water to cover the entire surface area of the walls a minimum

of 2 times. Transfer the rinse water into the narrow acid tank using the procedures described in Section 9 (see NOTE 4). Indirect pumping with the diaphragm pump should be used as a first choice.

NOTE 4: Since rinse water from acid baths is not to be placed into the "Acid Waste Bulk" drum, no liquid other than rinses must be present in the narrow acid tank. If acid waste liquid other than rinses are present in the narrow acid tank, then transfer this liquid into the "Acid Waste Bulk" drum prior to initiating acid bath cleaning procedures as described in Section 9.

10.1.3 Repeat the procedure described in 10.1.2 one more time (for a total of two times).

10.1.4 Prior to disposal, neutralize the acid tank rinse water in the narrow acid waste tank by adding a slurry of baking soda in water to the tank combined with mixing using a section PVC pipe (See Note 5). Use pH paper to monitor the pH during this neutralization process. Avoid adding excessive quantities of baking soda. After the pH has been raised to 6 or greater, empty the contents of the tank by opening the drain valve at the bottom of the narrow acid tank. Using the clean tygon hose connected to the DI water tap, rinse down the inside walls of the narrow acid tank to remove any nonreacted baking soda from the tank. Close the drain valve after rinsing the tank.

NOTE 5: Use of a slurry of baking soda is preferred over use of powder to avoid excessive sodium contamination of the acid bath hood areas. Make a slurry in a bucket well removed from glassware prep areas by mixing approximately equal amounts of baking soda and DI water. Use a plastic or glass stirring rod to mix the slurry.

10.2 New Acid Baths

Acid baths which have never been used for glassware preparation must be initially cleaned and scrubbed using soap and DI water. Follow the General Labware Cleaning Procedure in ASF-201 SOP as close as possible to prepare new acid baths. After initial cleaning, execute the rinsing procedures described in Section 10.1 prior to use.

APPENDIX O

PROTOCOL FOR SAMPLING HOUSEVAC EXHAUST EMISSIONS

PROTOCOL FOR SAMPLING Housevac EXHAUST EMISSIONS

1.0 INTRODUCTION

Part of the work on this study is directed to testing dust emissions in the air exhausted from each of the four brands of Housevacs. Dust concentrations and air flow rates will be determined in each test. There are a total of 12 tests, since each Housevac will be tested 3 times. One size class (< 53 μm) of test dust will be used in all tests.

2.0 EQUIPMENT AND SUPPLIES

- Turntable for dust feed
- Housevac enclosure and exhaust piping, etc., as shown in Figure O-1.
- Pitot tubes and inclined manometer or magnehelic gauge
- Particulate concentration monitor
- Strip chart recorder

3.0 PROCEDURE

3.1 Preparations for Testing Each Housevac

- Install new bag in Housevac and run 5 min.
- Remove bag; wait 5 min. then record tare weight of bag. Reinstall tared bag in Housevac.
- Place Housevac in enclosure. Push suction hose through opening in enclosure. Seal with duct tape if necessary.
- Insert pitot tube (and T/C) in 2 in. diameter exhaust duct.
- Turn on Housevac, with nozzle positioned well above turntable, and note ΔP reading for pitot tube in 2 in. diameter exhaust duct.

- Lower nozzle toward turntable to determine how close nozzle can be positioned without affecting pitot reading. Mark this position, and use it in conducting the tests described below.

- Record temperature and ΔP reading for pitot tube.

- Shut off Housevac. Calculate total flow rate in duct (see Table O-1).

- Select diameter of sampling nozzle, and/or required sampling rate, to provide isokinetic sampling for the particulate concentration monitor (see attached Table O-2). Adjust sampling rate, then install nozzle at center point of 8 in duct.

- Connect output signal from particulate concentration monitor to strip chart recorder.

- Weigh out 5 g. of sieved dust (<53 μm size). Distribute dust into shallow trough on turntable, as evenly as possible over the entire length of the trough circle, except for 6 inches on each side of the nozzle.

- Position nozzle of Housevac in marked position just above turntable and in the middle of the section of trough that does not contain dust.

3.2 Conduct Test

3.2.1 Initial Test

- Turn on particulate monitor and strip chart recorder. Mark date, time, and run number on strip chart. Also, identify each of the following steps on the strip chart, and times. Record data on data form (Table O-3).

- Turn on Housevac. Run for 1 min.

- Turn on the turntable, mark time when dust pickup begins.

- Continue running for 5 min., which should remove all dust from the turntable (i.e., one revolution).

- Continue running for 1 min., then stop test.

- Remove bag from Housevac; wait 5 min., then record weight of bag.

3.2.2 Repeat Test

- Install new bag in Housevac and run 5 min.
- Remove bag; wait 5 min. then record tare weight of bag. Reinstall tared bag in Housevac.
- Weigh out 5 g. of sieved dust (< 53 μm), and distribute evenly in trough, as before.
- Position nozzle above turntable.
- Conduct test as in Section 3.2.1.
- Repeat test a total of 3 times (total of 3 tests using the same Housevac).

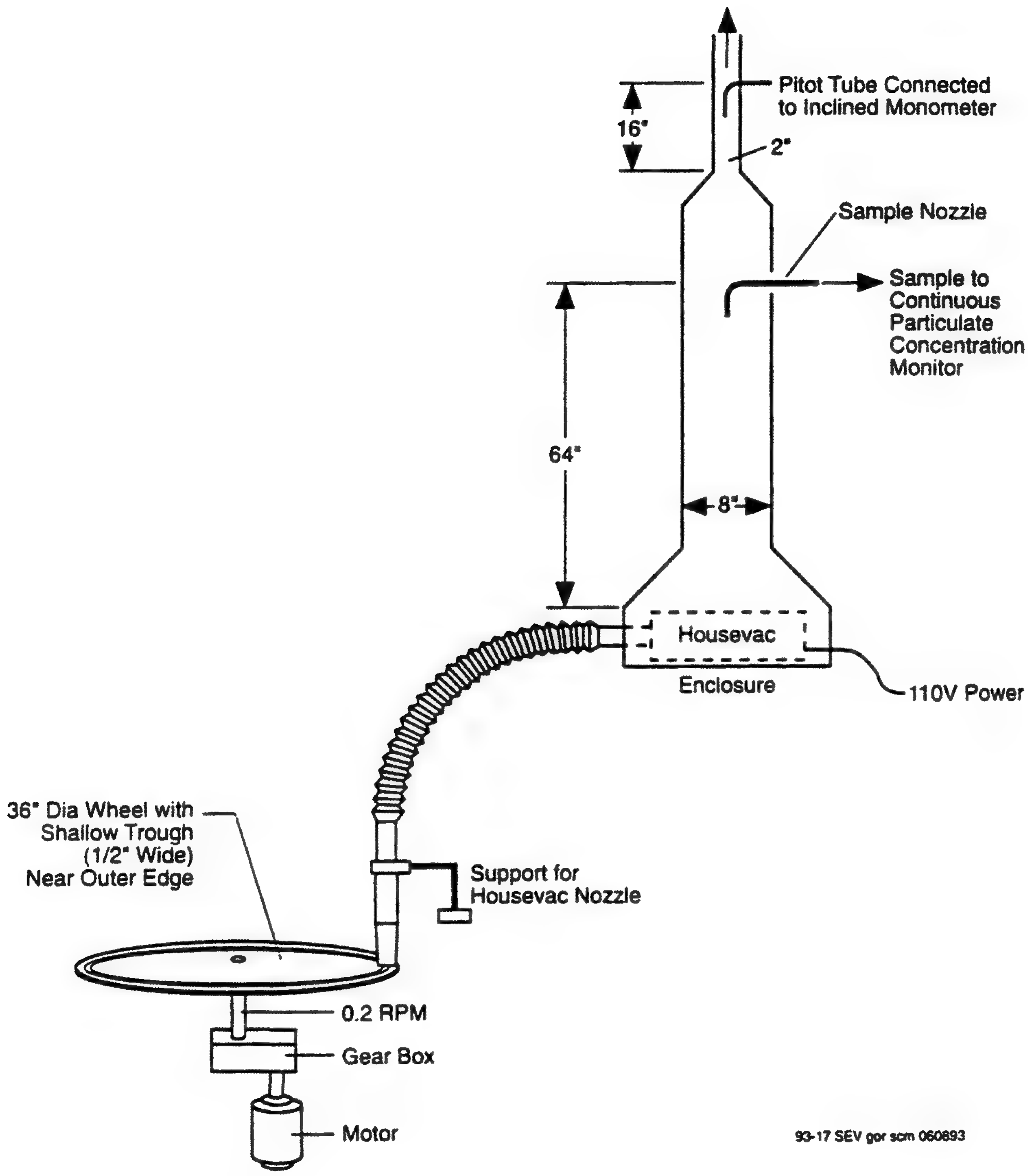

Figure O-1. Schematic diagram of test system for dust emissions testing.

4.0 COLLECTION OF SAMPLE

It would be desirable to use a research sampling vacuum to sample the exhaust dust emissions, for Pb analysis. However, it is expected that the dust concentration will be quite low, precluding this sampling. If preliminary testing indicates that sampling is feasible (and can be done isokinetically) it will be carried out using the most appropriate sampler and this protocol will be revised to include that sampling.

5.0 CONTAMINATION AVOIDANCE

For this protocol, contamination refers to the inadvertent increase (or decrease) in the weight of dust collected in the Housevac bag (other than that applied onto the turntable). To avoid this, the Housevac must be run only as specified in the protocol, to avoid "sucking in" any other dust in the vicinity. Care must especially be taken in removing the bag from the Housevac for weighing, so that none of the collected dust escapes or is allowed to fall out of the bag.

6.0 DEVIATIONS FROM PROTOCOL

Every attempt shall be made to follow this protocol. Deviations from the protocol may compromise the data quality and completeness objectives of the project. Deviations from the protocol will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual (or unanticipated conditions).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the testing data form and immediately notify the team leader and the MRI work assignment leader. Corrective action(s) shall be taken to ensure that the situation is not repeated. If possible, any tests affected by the inadvertent deviation shall be redone in accordance with the specified protocol.

Deliberate deviations from the protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be expedited. The MRI work assignment leader or the task leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals.

TABLE O-1
CALCULATION OF AIRFLOW RATE IN 2 IN. DUCT

Step 1 - Measurements

Measure pitot reading at center point, in inches of H_2O	$= \Delta P$
Measure temperature in duct in °F	$= t$
Record barometric pressure, in inches of Hg	$= P_B$

Step 2- Calculations

a. Calculate air density in lb/ft^3 $= \rho$

$$\rho = 1.325 \left(\frac{P_B}{460 + t} \right) \qquad \text{lb/ft}^3$$

b. Calculate velocity in ft/min (at center point)

$$V = 1{,}096 \sqrt{\frac{\Delta P}{\rho}} \qquad \text{ft/min}$$

c. Calculate average velocity in ft/min

$$V_{avg} = (V)(0.9) \qquad \text{ft/min}$$

d. Calculate airflow rate in acfm $= Q_E$

$$Q_E = (V_{avg})(0.0218 \text{ ft}^2) \qquad \text{acfm}$$

Convert airflow rate to acm/min

$$Q_M = Q_E(0.0283 \text{ m}^3/\text{ft}^3) \qquad \text{m}^3/\text{min}$$

Note: Airflow rate in m^3/min can be used to calculate dust emission in mg/min, based on dust concentration measured in mg/m^3. This assumes that dust concentration is measured at the same conditions of t and P_B as gas flow.

TABLE O-2
CALCULATION OF NOZZLE SIZE AND SAMPLING RATE NEEDED FOR ISOKINETIC SAMPLING OF DUST CONCENTRATION

Step 1 - Measurements

Measure pitot reading at center point of 2 in duct, in inches of H_2O	$= \Delta P$
Measure temperature in duct in °F	$= t$
Record barometric pressure, in inches of Hg	$= P_B$

Step 2 - Calculate velocity in 2 inch duct (V_2)

density of air in lb/ft^3 $= \rho = 1.325\left(\frac{P_B}{460 + t}\right)$ — Should be about 0.070 lb/ft^3

velocity in ft/min $= V_2 = 1{,}096 \sqrt{\frac{\Delta P}{\rho}}$ — Should be about 2,800 ft/min

Step 3 - Estimate velocity in 8 inch duct (V_8)

$V_8 = \frac{1}{16}(V_2)$ — Should be about 175 ft/min

Step 4 - Determine nozzle size required
(for sampling rate of 2 L/min = 0.070 ft^3/min)

d (in inches) $= \frac{3.61}{\sqrt{V_8}}$ — Should be about 0.27 in.

Select nearest nozzle size and determine if it is within 10 percent of actual size needed as calculated above. If actual size available is not within 10% of the required size as calculated above, select nearest larger nozzle size and go to Step 5.

TABLE O-2 (continued)

Step 5 - Determine sampling rate to be used
Determine V_8 per Step 3, in ft/min
Record d_{act} (i.e., actual nozzle size in inches)

Let S = sampling rate required in L/min

$$S = \frac{V_8 d_{act}^2}{6.47}$$

Should be in the range of 2 to 3 L/min

Step 6 - Adjust sampling rate

- Adjust the RAM-1 sampling rate to correspond to that determined above
- Adjust the RAM-1 purge rate to 10% of the sampling rate
- Shut off the RAM-1, and install sampling nozzle at center point of the 8 in. duct

TABLE O-3
DATA FORM FOR HOUSEVAC EMISSION TESTS

Date ______________
Operator ______________
Housevac _____ (A, B, C, or D)
Replicate _____ (Rep 1, 2, or 3)

Procedures
See attached pages

a. Preparations

Time	Bag tare weight (g)	RH (%)	Temp. (°F)	Bar. Press. (in. Hg)

2-in. duct pitot reading _________ in. of H_2O, and temp _________ °F
Calculated gas flow rate _________ acfm

Nozzle size selected ________ inches
Sample rate selected _________ L/min

Dust applied to turntable		
Total wt. (g)	Tare wt. (g)	Net wt. of dust (g)

b. Conduct Test (mark times on strip chart)

Operation	Time	2 in. duct ΔP rdg. (in H_2O)	Particulate conc. (mg/m^3)
Start housevac (run 1 min)			
Start turntable and lower the nozzle to near turntable			
1 min			
2 min			
3 min			
4 min			
5 min			
Stop housevac (run 1 min)			

Weigh Bag

Time	Bag final wt. (g)	RH (%)	Temp. (°F)

Project 9802
WA No. 55

**Data Entry Sheets
for
Housevac Tests**

Test Sequence Number ____________
Date ____________
Operator ____________

Test Identification

Housevac ________ (A, B, C or D)
Substrate ________ (TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in ________ (Yes, No)
Dust Amount ________ (100, 400 mg/ft^2
Pb Conc ________ (Low, High)
Dust Size ________ (<53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team ________ (number 1 or 2)

Procedure

Perform the tests according to the housevac test sequence in Appendix P, and vac procedure in Appendix I
Tare weigh new bag:
Run free 40 sec, cool 2 min, brush and record weight after 1 more min
Vacuum for 40 sec before any dust deposit
Reweigh bag (cool 2 min, brush and record weight after 1 more min)
Deposit dust, vacuum 40 sec, weigh the bag. Total of 3 times. (Grind-in after each dust deposit, if applicable)
Repeat vacuuming only (vacuum 40 sec, weigh the bag) 3 times
Shake dust from the bag, weigh, prepare for lead analysis
Vacuum dust from wand and brush (no weighing)

		Weight of Dust (Balance #________)				Weight of Bag (Balance #________)	
		Total Wt gm.	Tare Wt gm.	Net Wt gm.	Time	Weight gm.	Increase gm.
Tare weight of bag	.0					____	
Vacuum and weigh	.1				____	____	____
Add dust, vac & weigh	.2	____	____	____		____	____
Add dust, vac & weigh	.3	____	____	____		____	____
Add dust, vac & weigh	.4	____	____	____		____	____
Vacuum & weigh	.5					____	____
Vacuum & weigh	.6					____	____
Vacuum & weigh	.7					____	____
Dust sent to lab	.8	____	____	____			

Bar Code for Sample	Bar Code for Blank

Submit one blank for each week

Sample relinquished by ____________
Sample received by ____________
Date of transfer ____________

Reviewed by ____________
Date reviewed ____________

APPENDIX P

TEST PATTERNS, HOUSEVACS

Substrate	Particle Size	Dust Loading	Lead Conc	Team	vacuum	Original Number	Revised Number
Linoleum	53-106	400 mg/sqft	High	Team 1	A	1001	
Linoleum	<53	100 mg/sqft	High	Team 1	D	1002	
Linoleum	212-250	100 mg/sqft	High	Team 1	C	1003	
Linoleum	106-150	100 mg/sqft	Low	Team 1	B	1004	
Wood	150-212	400 mg/sqft	High	Team 1	A	1005	
Wood	106-150	400 mg/sqft	Low	Team 1	B	1006	
Wood	150-212	400 mg/sqft	High	Team 1	C	1007	1-28
Wood	<53	100 mg/sqft	Low	Team 1	D	1008	
Carpet	53-106	400 mg/sqft	Low	Team 1	D	1009	1-10
Carpet	53-106	400 mg/sqft	Low	Team 1	A	1010	
Carpet	53-106	400 mg/sqft	Low	Team 1	C	1011	
Carpet	53-106	400 mg/sqft	Low	Team 1	B	1012	
Carpet	<53	100 mg/sqft	Low	Team 1	C	1013	
Carpet	212-250	100 mg/sqft	Low	Team 1	D	1014	
Upholstery	<53	100 mg/sqft	High	Team 1	B	1020	1-1
Upholstery	<53	100 mg/sqft	High	Team 1	C	1085	1-2
Upholstery	212-250	100 mg/sqft	High	Team 1	B	1088	1-3
Carpet w Grind-in	212-250	100 mg/sqft	High	Team 1	A	1052	1-4
Carpet w Grind-in	212-250	100 mg/sqft	High	Team 1	B	1101	1-5
Carpet w Grind-in	<53	100 mg/sqft	High	Team 1	D	1104	1-6
Upholstery	53-106	400 mg/sqft	High	Team 1	D	1030	1-7
Upholstery	53-106	400 mg/sqft	High	Team 1	A	1032	1-8
Carpet	106-150	100 mg/sqft	High	Team 1	A	1046	1-9
Linoleum	212-250	100 mg/sqft	High	Team 1	D	1026	1-11
Linoleum	106-150	100 mg/sqft	Low	Team 1	C	1027	1-12
Linoleum	150-212	400 mg/sqft	Low	Team 1	B	1056	1-13
Carpet	<53	100 mg/sqft	Low	Team 1	A	1015	1-14
Carpet	212-250	100 mg/sqft	Low	Team 1	A	1083	1-15
Carpet w Grind-in	53-106	400 mg/sqft	High	Team 1	C	1069	1-16
Linoleum	<53	100 mg/sqft	High	Team 1	B	1075	1-17
Linoleum	53-106	400 mg/sqft	High	Team 1	C	1076	1-18
Linoleum	250-2000	400 mg/sqft	High	Team 1	B	1095	1-19
Upholstery	150-212	400 mg/sqft	Low	Team 1	B	1042	1-20
Upholstery	150-212	400 mg/sqft	Low	Team 1	A	1044	1-21
Carpet	250-2000	400 mg/sqft	High	Team 1	B	1065	1-22
Carpet	150-212	400 mg/sqft	High	Team 1	D	1119	1-23
Carpet w Grind-in	150-212	400 mg/sqft	Low	Team 1	B	1038	1-24
Wood	212-250	100 mg/sqft	Low	Team 1	B	1079	1-25
Wood	106-150	400 mg/sqft	Low	Team 1	D	1080	1-26
Wood	250-2000	100 mg/sqft	High	Team 1	A	1111	1-27
Wood	53-106	400 mg/sqft	Low	Team 1	B	1058	1-29
Wood	<53	100 mg/sqft	Low	Team 1	C	1077	1-30

Substrate	Particle Size	Dust Loading	Lead Conc	Team	vacuum	Original Number	Revised Number
Tile	150-212	100 mg/sqft	Low	Team 2	C	2001	
Tile	212-250	400 mg/sqft	High	Team 2	B	2002	
Tile	150-212	100 mg/sqft	Low	Team 2	A	2003	
Tile	<53	400 mg/sqft	High	Team 2	D	2004	
Carpet	212-250	400 mg/sqft	High	Team 2	C	2005	
Carpet	<53	400 mg/sqft	High	Team 2	D	2006	
Carpet	<53	400 mg/sqft	High	Team 2	A	2007	
Carpet	212-250	400 mg/sqft	High	Team 2	B	2008	
Carpet w Grind-in	212-250	400 mg/sqft	Low	Team 2	C	2009	
Carpet w Grind-in	212-250	400 mg/sqft	Low	Team 2	B	2010	
Carpet w Grind-in	<53	400 mg/sqft	Low	Team 2	D	2011	
Carpet w Grind-in	<53	400 mg/sqft	Low	Team 2	A	2012	2-1
Linoleum	53-106	100 mg/sqft	Low	Team 2	A	2013	
Linoleum	150-212	100 mg/sqft	High	Team 2	B	2014	
Carpet w Grind-in	212-250	400 mg/sqft	Low	Team 2	D	2098	2-2
Carpet w Grind-in	<53	400 mg/sqft	Low	Team 2	C	2099	2-3
Linoleum	250-2000	100 mg/sqft	Low	Team 2	C	2033	2-4
Linoleum	<53	400 mg/sqft	Low	Team 2	A	2048	2-5
Linoleum	53-106	100 mg/sqft	Low	Team 2	D	2061	2-6
Carpet	53-106	100 mg/sqft	High	Team 2	C	2106	2-7
Upholstery	53-106	100 mg/sqft	Low	Team 2	C	2025	2-8
Wood	212-250	400 mg/sqft	High	Team 2	C	2065	2-9
Wood	53-106	100 mg/sqft	High	Team 2	A	2068	2-10
Wood	<53	400 mg/sqft	High	Team 2	D	2095	2-11
Carpet w Grind-in	150-212	100 mg/sqft	High	Team 2	A	2109	2-12
Carpet w Grind-in	150-212	100 mg/sqft	High	Team 2	C	2112	2-13
Wood	106-150	100 mg/sqft	High	Team 2	B	2018	2-14
Wood	150-212	100 mg/sqft	Low	Team 2	A	2020	2-15
Wood	250-2000	400 mg/sqft	Low	Team 2	D	2052	2-16
Linoleum	212-250	400 mg/sqft	Low	Team 2	A	2064	2-17
Linoleum	150-212	100 mg/sqft	High	Team 2	D	2085	2-18
Linoleum	106-150	400 mg/sqft	High	Team 2	A	2088	2-19
Carpet w Grind-in	53-106	100 mg/sqft	Low	Team 2	D	2043	2-20
Carpet w Grind-in	53-106	100 mg/sqft	Low	Team 2	B	2044	2-21
Carpet	212-250	400 mg/sqft	High	Team 2	D	2058	2-22
Carpet	<53	400 mg/sqft	High	Team 2	B	2060	2-23
Upholstery	<53	400 mg/sqft	Low	Team 2	D	2089	2-24
Upholstery	212-250	400 mg/sqft	Low	Team 2	C	2091	2-25
Upholstery	212-250	400 mg/sqft	Low	Team 2	A	2101	2-26
Upholstery	150-212	100 mg/sqft	High	Team 2	D	2078	2-27
Carpet	106-150	400 mg/sqft	Low	Team 2	C	2071	2-28
Carpet	150-212	100 mg/sqft	Low	Team 2	B	2022	2-29
Carpet	250-2000	100 mg/sqft	Low	Team 2	C	2074	2-30

APPENDIX Q

TEST PATTERN, SAMPLERS

Substrate	Particle Size	Dust Loading	Lead Conc	Team	Vacuum	Square	Revised Number
Carpet	53-106	400 mg/sqft	Low	Team 1	R&M HVS3	4	3-1
Upholstery	<53	100 mg/sqft	High	Team 1	CAPS cyclone	2	3-2
Upholstery	<53	100 mg/sqft	High	Team 1	Blue nozzle	3	3-3
Upholstery	212-250	100 mg/sqft	High	Team 1	Blue nozzle	4	3-4
Upholstery	150-212	400 mg/sqft	Low	Team 1	Blue nozzle	3	3-5
Wood	106-150	400 mg/sqft	Low	Team 1	R&M HVS3	2	3-6
Wood	53-106	400 mg/sqft	Low	Team 1	Blue nozzle	3	3-7
Carpet	150-212	400 mg/sqft	High	Team 1	R&M HVS3	1	3-8
Carpet	250-2000	400 mg/sqft	High	Team 1	Blue nozzle	2	3-9
Carpet w Grind-in	53-106	400 mg/sqft	High	Team 1	CAPS cyclone	3	3-10
Carpet w Grind-in	150-212	400 mg/sqft	Low	Team 1	Blue nozzle	1	3-11
Wood	150-212	400 mg/sqft	High	Team 1	CAPS cyclone	4	3-12
Linoleum	106-150	100 mg/sqft	Low	Team 1	CAPS cyclone	2	3-13
Wood	212-250	100 mg/sqft	Low	Team 1	Blue nozzle	1	3-14
Wood	<53	100 mg/sqft	Low	Team 1	CAPS cyclone	2	3-15
Upholstery	53-106	400 mg/sqft	High	Team 1	R&M HVS3	3	3-16
Linoleum	250-2000	400 mg/sqft	High	Team 1	Blue nozzle	1	3-17
Linoleum	53-106	400 mg/sqft	High	Team 1	CAPS cyclone	2	3-18
Carpet w Grind-in	212-250	100 mg/sqft	High	Team 1	Blue nozzle	2	3-19
Carpet w Grind-in	<53	100 mg/sqft	High	Team 1	R&M HVS3	3	3-20
Linoleum	<53	100 mg/sqft	High	Team 1	Blue nozzle	3	3-21
Linoleum	212-250	100 mg/sqft	High	Team 1	R&M HVS3	4	3-22
Linoleum	150-212	400 mg/sqft	Low	Team 1	Blue nozzle	4	3-23
Wood	250-2000	100 mg/sqft	High	Team 1	Baby Wipe	4	3-24

Substrate	Particle Size	Dust Loading	Lead Conc	Team	Vacuum	Square	Revised Number
Linoleum	212-250	400 mg/sqft	Low	Team 2	Baby Wipe	1	4-1
Linoleum	<53	400 mg/sqft	Low	Team 2	Baby Wipe	2	4-2
Carpet	53-106	100 mg/sqft	High	Team 2	CAPS cyclone	1	4-3
Carpet	<53	400 mg/sqft	High	Team 2	Blue nozzle	3	4-4
Carpet	212-250	400 mg/sqft	High	Team 2	R&M HVS3	4	4-5
Carpet w Grind-in	<53	400 mg/sqft	Low	Team 2	CAPS cyclone	1	4-6
Carpet w Grind-in	212-250	400 mg/sqft	Low	Team 2	R&M HVS3	2	4-7
Linoleum	106-150	400 mg/sqft	High	Team 2	Baby Wipe	4	4-8
Upholstery	53-106	100 mg/sqft	Low	Team 2	CAPS cyclone	4	4-9
Upholstery	<53	400 mg/sqft	Low	Team 2	R&M HVS3	1	4-10
Upholstery	212-250	400 mg/sqft	Low	Team 2	CAPS cyclone	2	4-11
Carpet w Grind-in	53-106	100 mg/sqft	Low	Team 2	Blue nozzle	1	4-12
Carpet w Grind-in	53-106	100 mg/sqft	Low	Team 2	R&M HVS3	2	4-13
Wood	150-212	100 mg/sqft	Low	Team 2	Baby Wipe	2	4-14
Linoleum	150-212	100 mg/sqft	High	Team 2	R&M HVS3	1	4-15
Carpet w Grind-in	150-212	100 mg/sqft	High	Team 2	CAPS cyclone	1	4-16
Linoleum	250-2000	100 mg/sqft	Low	Team 2	CAPS cyclone	2	4-17
Linoleum	53-106	100 mg/sqft	Low	Team 2	R&M HVS3	3	4-18
Carpet	250-2000	100 mg/sqft	Low	Team 2	CAPS cyclone	1	4-19
Carpet	150-212	100 mg/sqft	Low	Team 2	Blue nozzle	2	4-20
Wood	<53	400 mg/sqft	High	Team 2	R&M HVS3	2	4-21
Wood	212-250	400 mg/sqft	High	Team 2	CAPS cyclone	3	4-22
Upholstery	150-212	100 mg/sqft	High	Team 2	R&M HVS3	3	4-23
Wood	53-106	100 mg/sqft	High	Team 2	Baby Wipe	2	4-24
Wood	106-150	100 mg/sqft	High	Team 2	Blue nozzle	3	4-25
Wood	250-2000	400 mg/sqft	Low	Team 2	R&M HVS3	2	4-26
Carpet	106-150	400 mg/sqft	Low	Team 2	CAPS cyclone	3	4-27

APPENDIX R

PROTOCOL FOR CONDUCTING TESTS WITH SAMPLERS

PROTOCOL FOR CONDUCTING TESTS WITH SAMPLERS

1.0 INTRODUCTION

Many of the tests to be done for this study require sampling with different types of samplers, each of which involves a specific protocol per Appendices E, F, G or H. These tests are to be conducted in accordance with the test pattern specified in Appendix Q, which includes designated "test squares" on each substrate, where samplers are to be tested (i.e., square 1, 2, 3 or 4). Moreover, the test design requires vacuuming with Housevac A before the first specified square or after the last specified square, by either team. Thus, the purpose of this protocol is to define the procedure that is to be utilized for all sampler tests, in conjunction with the specific procedures for each sampler in Appendices E, F, G or H.

2.0 EQUIPMENT AND SUPPLIES

Housevac A
Template (1 ft^3)
Other equipment listed for each sampler in Appendices E, F, G or H.

3.0 PROCEDURE

3.1 Preparations

- When substrate is first used, mark four 1 ft^2 squares within the already marked Housevac test area.
- Outside the test area, number each square as 1, 2, 3, and 4. (1, 2, and 3 on carpet and upholstery).

3.2 CONDUCT TEST

- Record all test data on data entry sheet shown at the end of this appendix.

- Refer to Appendix Q to determine the parameters for the test to be performed, which includes the sampler to be used and test square to be used.

- If first square:

  - Tare weigh Housevac bag (run free 40 seconds, cool 2 minutes, brush and record weight after 1 more minute).

  - Vac the entire test area for 40 seconds with Housevac A.

  - Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute).

- Deposit specified dust within test square, and determine amount deposited.

- Grind in dust, if applicable.

- Sample dust according to protocol for sampler to be tested (Appendices E, F, G, or H).

- Prepare dust sample for analysis per sampler protocol.

- If the square used for the sampler test was the last square, vacuum the entire test area with Housevac A after the sampler test:

  - Tare weigh Housevac bag (run free for 120 seconds, cool 2 minutes, brush and record weight after 1 more minute).

  - Vac the entire test area for 120 seconds with Housevac A.

  - Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute).

- Vacuum dust from wand and brush of Housevac A (no weighing)

4.0 CONTAMINATION AVOIDANCE

Contamination refers to both the inadvertent increase (or decrease) in the weight of dust collected by the Housevac or sampler (other than that applied to the test square) and potential Pb concentration of the sampler dust sample.

Care must be taken to prevacuum only the test square. Also, great care will be needed to distribute the dust only on the test square, and avoid transfer of dust to or from other test squares when grind-in of the dust is required.

5.0 DEVIATIONS FROM PROTOCOL

Every attempt shall be made to follow this protocol. Deviations from the protocol may compromise the data quality and completeness objectives of the project. Deviations from the protocol will generally fall into two categories: inadvertent deviations (procedural errors) and deliberate deviations (modifications to the protocol in response to unusual or unanticipated conditions).

In the case of inadvertent deviations from the protocol, the sampling team shall fully document the deviation on the sampling data form and immediately notify the MRI work assignment leader. Corrective action(s) shall be given to ensure that the situation is not repeated. If possible, any tests or samples affected by the inadvertent deviation shall be redone in accordance with the specified protocol.

Deliberate deviations from the protocol should be approved in advance with a signed modification to the QAPjP. If time is critical, preliminary verbal approval may be granted by EPA. These verbal approvals will be followed up with a signed modification to the QAPjP. In either case, the sampling team should notify all parties concerned in a timely manner so that the approval mechanism can be expedited. The MRI work assignment leader is responsible for initiation of the QAPjP modification and acquiring the necessary approvals.

Data Entry Sheets
for
Sampler Tests

Test Sequence Number ____________
Date ________________________
Operator ______________________

Test Identification

Sampler	_____	(Blue Nozzle, CAPS, HVS3 or WIPE)
Substrate	_____	(TILE, LINOleum, WOOD, UPHOlstery, CaRPeT)
Grind-in	_____	(Yes, No)
Dust Amount	_____	(100, 400 mg/ft^2)
Pb Conc	_____	(Low, High)
Dust Size	_____	(>53, 53-106, 106-150, 150-212, 212-250, 250-2000)
Team	_____	(Number 1 or 2)
Square number	_____	(1, 2, 3 or 4) 1=first, 3=last for carpet and upholstery, else 4=last)

Procedure

Perform the tests according to the sampler test sequence in Appendix Q, and procedures in Appendix E, F, G or H. Housevac A will be used to vacuum the first square before sampler tests, or to vacuum the last square after sampler tests.

If first square:
- Tare weigh bag (run free for 40 seconds, cool 2 minutes, brush and record weight after 1 more minute)
- Vac square for 40 seconds with Housevac A
- Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)

Deposit dust in specified square and weigh the amount deposited (Grind-in dust if applicable)
Sample dust according to the appropriate protocol, weigh the dust collected (except for wipes)
Prepare the dust sample for analysis
If last square:
- Tare weigh bag (run free for 120 seconds, cool 2 minutes, brush and record weight after 1 more minute)
- Vac square for 120 seconds with Housevac A
- Reweigh bag (cool 2 minutes, brush and record weight after 1 more minute)

Vacuum dust from wand and brush (no weighing)

	Weight of Dust (Balance #________)				Weight of Bag (Balance #________)	
	Total Wt. gm.	Final Wt. gm.	Net Wt. gm.	Time	Weight gm.	Increase gm.
Initial weight of bag (if first or last square)	.0				_______	
Vacuum and reweigh bag (if first square)	.1				_______	_______
Dust deposited	.2_____	_______	_______	_______		
Dust collected by sampler (exclu wipes)	.3_____	_______	_______			
Vacuum & reweigh bag (if last square)	4.				_______	_______

Bar Code for Sample	Bar Code for Blank

NOTE: Submit one blank for each sampler, once each week

Sample relinquished by ________________ Reviewed by ________________
Sample received by ________________ Date reviewed ________________
Date of transfer ________________

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16. Abstract (Limit: 200 words)

This document reports the results of a laboratory study to evaluate dust and lead recovery of four house dust sampling methods used in previous EPA studies and four commercially available household vacuums. The dust sampling methods included those used in the Baltimore Repair and Maintenance study (BRM), the Comprehensive Abatement Performance Study (CAPS), the HUD National Survey of Lead-Based Paint (Blue Nozzle sampler), and the HUD's wipe sampling method. The testing protocols used house dust from homes built before 1963 and after 1982, sieved into six particle size classes ranging from 0 to 2,000 microns and applied to five substrates. Dust from older dwellings had a higher lead concentration than the dust from newer homes. The BRM and the CAPS samplers produced the highest dust recoveries across all substrates and particle sizes. The Blue Nozzle sampler had the lowest recoveries. The pattern of lead recovery across samplers was similar to dust recovery, with the wipe lead recovery similar to that for the CAPS cyclone. Exhaust tests showed that 0.02% or less of the dust passed through the vacuum cleaner bag. Adjustments to the results from the HUD National Survey of Lead-Based Paint are also discussed.

17. Document Analysis a. Descriptors

Environmental Contaminants

b. Identifiers/Open-Ended Terms

Dust lead, regression analysis, National Survey of Lead-Based Paint, dust samplers, vacuum cleaners

c. COSATI Field/Group

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